University of São Paulo
"Luiz de Queiroz" College of Agriculture

Organomineral fertilizer from sewage sludge: nutrient recycling and environmental safety for tropical agriculture

Mayra Maniero Rodrigues

Thesis presented to obtain the degree of Doctor in Science:
Area: Soil and Plant Nutrition

Piracicaba
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versão revisada de acordo com a resolução CoPGr 6018 de 2011

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Prof. Dr. **JUSSARA BORGES REGITANO**

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Francisco de Assis Maniero (*In memoriam*)

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EPIGRAPH

"The future, at least, had the advantage of not being the present, and the worse can always take a turn for the better... For, strange though it may seem, she had faith."
Clarice Lispector

"Nothing in life is to be feared, only understood. Now it's time to understand more to fear less."
Marie Curie

"Nothing is absolute. Everything changes, everything moves, everything spins, everything flies and disappears."
Frida Kahlo

"Farming is no longer a profession, but a passion for nature, for true life, for being born, growing, blossoming and maturing."
Ana Maria Primavesi

"I dream of a country where education will prevail."
Malala Yousafzai

CONTENTS

RESUMO

Fertilizante organomineral de lodo de esgoto: reciclagem de nutrientes e segurança ambiental para a agricultura tropical

A disposição adequada de lodo de esgoto (LE) é um dos grandes desafios para a sustentabilidade da sociedade moderna. Este material é rica fonte de matéria orgânica e nutrientes para as plantas. Contudo, seu uso na agricultura está associado a elevadas concentrações de metais pesados como Zn e Ni, em níveis que podem inviabilizar sua aplicação no solo. Nesse sentido, o uso de LE na síntese de fertilizantes organominerais (FOM-LE) é uma estrategia promissora que permite alinhar o descarte adequado de LE à melhorias na fertilidade dos solos e redução da dependencia por fertilizantes minerais (FMs), principalmente na agricultura tropical. Nosso objetivo foi explorar os efeitos de uma formulação FOM-LE em solo-planta-microrganismos, a fim de compreender possíveis efeitos sinérgicos e inibitorios da mistura mineral e orgânica sobre agroecossistemas tropicais. Uma revisão crítica de literatura baseada em análise bibliométrica foi realizada para compreender o estado da arte sobre FOM-LEs entre 2012-2023, potencialidades e lacunas sobre a sua sintese e utilização como alternativa ao uso exclusivo de LE em cultivos. A agricultura tropical tem grande potencial para utilização deste fertilizante, como o Brasil, devido a presença de solos intemperizados, com baixos teores de matéria orgânica e micronutrientes, os quais demandam grandes quantidades de insumos externos. Em seguida, propusemos a síntese e caracterização de um FOM-LE 4-8-8 em três formas físicas (farelo, grânulo e pellet) e avaliamos o seu potencial agronômico e segurança ambiental. O produto final obtido foi livre de agentes patogênicos, teores seguros de metais pesados, e atendeu aos requisitos estabelecidos pela legislação para uso agrícola. Um estudo de campo foi elaborado no qual o FOM-LE, nas três formas físicas, foi aplicado em duas doses (70% e 100% da necessidade da soja de P_2O_5) e comparado a um FM, em sistema de sucessão soja-milho safrinha em plantio direto em área de Cerrado brasileiro. Comparado ao LE, a dose de aplicação de FOM-LE é 3,5 vezes menor, devido a maior concentração de nutrientes, o que favorece operações de transporte e aplicação no campo. Nossos principais achados indicam que, no primeiro ano de aplicação na soja, o FOM-LE favorece a nodulação das plantas, principalmente grânulo e pellet, comparado ao uso de FM. Independentemente da forma física, respostas agronômicas, teores de metais pesados em solo-planta e efeitos sobre bioindicadores de qualidade do solo, foram equivalentes entre FOM-LE e FM, tanto para soja quanto para o milho safrinha. O efeito residual no milho safrinha foi maior com o uso de dose equivalente ao FM, sendo interessante a forma física peletizada. Nós acreditamos que essa abordagem holistica encoraja estratégias sustentáveis de manejo na agricultura tropical, combinando sustentabilidade ambiental e ganhos produtivos.

Palavras-chave: Sustentabilidade; Resíduo orgânico; Biossólido; Biofertilizante

ABSTRACT

Organomineral fertilizer from sewage sludge: nutrient recycling and environmental safety for tropical agriculture

The proper disposal of sewage sludge (SS) is one of the great challenges for the sustainability of modern society. This material is a rich source of organic matter and nutrients for plants. However, its use in agriculture is associated with high concentrations of heavy metals such as Zn and Ni, at levels that may make its application in the soil unfeasible. In this sense, the use of SS in the synthesis of organomineral fertilizers (SS-OMF) is a promising strategy that allows aligning the proper disposal of SS with improvements in soil fertility and reduction of dependence on mineral fertilizers (MFs), mainly in tropical agriculture. Our objective was to explore the effects of an SS-OMF formulation on soil-plant-microorganisms and to understand possible synergistic and inhibitory effects of the mineral and organic mixture on tropical agroecosystems. A critical literature review based on bibliometric analysis was carried out to understand the state of the art on SS-OMFs between 2012-2023, potentialities, and gaps regarding its synthesis and use as an alternative to the exclusive use of SS in crops. Tropical agriculture has great potential for using this fertilizer, in Brazil, due to the presence of weathered soils, with low levels of organic matter and micronutrients, which demand large amounts of external inputs. Then, we proposed the synthesis and characterization of an SS-OMF 4-8-8 in three physical forms (powder, granule, and pellet) and evaluated its agronomic potential and environmental safety. The final product obtained was free of pathogenic agents, had safe levels of heavy metals, and met the requirements established by legislation for agricultural use. A field study was carried out in which the SS-OMF, in the three physical forms, was applied at two rates (70% and 100% of the P_2O_5 soybean requirement) and compared to an MF in a soybean maize off-season succession system in no-tillage in the Brazilian Cerrado area. Compared to SS, the application rate of SS-OMF is 3.5 times lower, due to the higher concentration of nutrients, which favors transport operations and application in the field. Our main findings indicate that, in the first year of application in soybeans, SS-OMF favors plant nodulation, mainly granules, and pellets, compared to the use of powder and MF. Regardless of the physical form, agronomic responses, heavy metal contents in soil-plant, and effects on soil quality bioindicators were equivalent between SS-OMF and MF, both for soybean and maize. The residual effect on maize off-season was greater with the use of an equivalent rate to MF, with the pellet's physical form being interesting. We believe that this holistic approach encourages sustainable management strategies in tropical agriculture, combining environmental sustainability and productivity gains.

Keywords: Sustainability; Organic waste; Biosolid; Biofertilizer

1. INTRODUCTION

On November 15, 2022, newspapers around the world reported a historic milestone in human development: the world population is now 8 billion people on Earth (United Nations 2022). Despite these data indicating the consolidation of the success of the human species on the planet, the pressure on natural resources for the adequate supply of modern society has never been so intense (Ramankutty et al. 2018; Rorat et al. 2019). It is estimated that about 60% of people live in urban centers in the world, occupying a space of 3% of the world's territory (Ritchie and Roser 2018). Consequently, health issues become increasingly sensitive as there is a need to manage the disposal of urban waste, such as sewage sludge (SS).

The raw SS generated by wastewater treatment plants (WWTPs) is the result of biochemical processes capable of generating a by-product with simpler organic components, organic matter (OM), and reduced amounts of pathogenic agents (Raheem et al. 2018; Rorat et al. 2019). It is estimated that the annual global production of SS is about 45 million tons (dry matter) generated in 60 thousand WWTPs operating around the world (di Giacomo and Romano 2022). When dry, this material has an average of 50-70% OM, 3-4% N, 0.5-2.5% P, and other nutrients to plants such as Zn and Cu, which makes this residue have great potential for use in agriculture (Alvarenga et al. 2015). However, it also contains potentially toxic inorganic elements such as the heavy metals Cr, Cd, Cu, Co, Hg, Ni, and Pb, pathogenic agents such as E. Coli and *Salmonella*, and persistent organic pollutants (POPs), which makes its use in soil fertilization an activity that must be done with caution (Cieślik et al. 2015; Sundha et al. 2022). Several countries such as Poland, France, Belgium, Sweden, and Germany have prohibited or restricted the disposal of SS in landfills, leading new research seeking environmentally safe alternatives for the use of SS in agriculture (Regitano et al. 2022).

On the other hand, the pressure for high agricultural productivity aligned with the intensive use of mineral fertilizers, pesticides, and non-conservative soil techniques are causes of environmental degradation, climate change, and loss of biodiversity in the world (Foley et al. 2011). At the global level, the agricultural sector contributes about 15% of methane (CH_4) and nitrous oxide (N_2O) emissions, mainly leveraged using nitrogen fertilizers and inadequate management of crops (Malhi et al. 2021). To mitigate such effects, approaches that enable nutrient recycling, OM input, and maintenance of soil microbial activity and diversity, have been increasingly incorporated into the production system, such as the use of organic wastes, such as SS (Verma and Verma 2012; Sharma et al. 2019).

The use of SS in agriculture is the subject of several studies, whether in the form of compost (Kummer et al. 2016; Bożym and Siemiątkowski 2018; Stacey et al. 2019), ash (Donatello and Cheeseman 2013; Antonkiewicz et al. 2020), *in natura* (Brandani et al. 2019), and more recently, in the form of organomineral fertilizers (OMFs) (Antille et al. 2013; Kominko et al. 2017; Smith et al. 2020). The OMFs are a viable and eco-friendly alternative for agricultural production, defined as the result of mixing or combining simple mineral fertilizers (MFs) and organic fractions, such as SS (Smith et al. 2020). The use of OMFs from the SS (SS-OMFs) allows the proper SS disposal instead of landfills, recycling of nutrients and OM, adjustment of the formulation to the nutritional needs of the crops, as well as the dilution of heavy metals quantity which, in adequate amounts, are considered micronutrients and beneficial for plants, such as Cu, Co, Mo, Zn, and Ni (Kominko et al. 2022). Compared to MFs, the use of SS-OMFs presents the slow release of nutrients from the organic fraction as it is mineralized, mainly N and P (Antille et al., 2014a; Antille et al., 2014b).

All these characteristics have shown that the use of SS-OMFs in tropical agriculture is a promising alternative for reducing external dependence on MFs, one of the major obstacles to food production in countries such as Brazil (Gomes et al. 2019). In general, the main challenges of Brazilian agriculture are the high P fixation in oxidic soils due to the high levels of Fe, Mn, and Al oxyhydroxides, K and B leaching due to the predominance of 1:1 clays and low OM added to periods of intense rain, depletion of stocks of micronutrients in the soil such as B, Mn, and Zn whose natural deficiency in crops is intensified by the use of formulations containing only NPK (Maranguit et al. 2017). For example, Zn deficiency is considered a limiting factor for plant development in the central region of the country, aggravated by using high doses of limestone and the practice of phosphating, making it less available in the soil solution (Nogueira et al. 2019) whose deficiency can be overcome using SS-OMF (Kominko et al., 2017). These natural characteristics mean that more than 50% of phosphate and 85% of potassium fertilizers in Brazil are imported, evidencing the country's high dependence on mineral sources of fertilizers (Pavinato et al. 2020; Sipert et al. 2020). All these factors enhance the benefits of using SS-OMFs in regions such as the Brazilian Cerrado, mainly composed of medium to low-fertility soils (Lemes et al. 2019; Soterroni et al. 2019).

Brazil is one of the main producers and exporters of food in the world, being the first in the production of soybean (153 million t^{-1}), a crop that occupies about 43.4 million hectares, and third about maize (125 million t^{-1}), spread over 21.5 million hectares (Conab 2023). The Cerrado is the biome that concentrates around 50% of the production of these two commodities, however, production costs with fertilizers are around 1/3 in both crops (Gomes et al. 2019; Aprosoja 2023). On the other hand, the country is estimated to generate 372,000 metric tons (dry basis) of SS annually (Mateo-Sagasta et al. 2015). This amount tends to increase after the recent enactment of the Basic Sanitation Legal Framework, increasing pressure for the universalization of basic sanitation, the generation of SS, and over landfills, the main form of disposal of this waste in Brazil (Ghini et al. 2016).

The adoption of practices that advocate the addition of organic materials, such as the use of SS-OMFs can promote benefits related to physical-chemical processes in tropical soils, such as competition for P adsorption sites by low molecular weight organic acids and humic and fulvic acids (Wu et al. 2013; Yu et al. 2013), decreasing its immobilization in the soil, making it more available for uptake by plants (Fink et al. 2016). Field studies in tropical soils are still incipient, however, they have indicated similar agronomic performance between SS-OMFs and MFs in soybean and sugarcane crops (Mota et al. 2019; Silva et al. 2020). In Brazil, the segment of special fertilizers has been growing and showed an increase of 80.5% in revenues in 2020, that is, around R$ 1.9 billion, mainly in the states of São Paulo, Minas Gerais and Mato Grosso (Abisolo 2023).

As important as the rates, the physical forms of presentation of SS-OMFs influence their final price and, consequently, their use by farmers (Fadare et al. 2010). The most used forms are powder, granules, and pellets, the last two being the most expensive (Romano et al. 2014). However, it is still unclear whether the physical forms are determinant in the release and use of nutrients by plants or the residual effect of SS-OMFs (Fachini et al. 2021), which would allow its use by subsequent crops, as is the case with maize off-season, cultivated shortly after the soybean harvest in central Brazil (Mieldazys et al. 2017; Pampuro et al. 2017). This understanding is also necessary when using SS with high heavy metals content once it is not clear whether the bioavailability of elements such as Ni and Zn in the soil solution is influenced by the SS-OMF particle arrangement (Rengel 2015; Fink et al. 2016; Mielki et al. 2016).

In addition to the physicochemical aspects of soils, there are gaps regarding possible synergistic or inhibitory effects of the mixture between the organic fraction (SS) and the mineral fraction of SS-OMF on bioindicator of soil quality, involved in ecosystem services related to plant nutrition, which may reflect in productive gains (Utobo and Tewari 2015; Jacoby et al. 2017). To understand changes in soil quality, bioindicators have been used to compare different forms of agroecosystem management, as is the case with the use of SS-OMFs. These bioindicators describe organisms that perform key functions in the soil ecosystem (Schloter et al. 2018). The most used are the activity of enzymes β-glucosidase (carbon cycle), acid phosphatase (P cycle), arylsulfatase (S cycle), quantification of easily extractable glomalin (EEG) (associated with the presence of arbuscular mycorrhizal fungi), and quantification of associated carbon the microbial biomass (CBM) (Ferraz-Almeida et al. 2015; Adetunji et al. 2017; Lehmann et al. 2020).

It is known that microbial activity plays a key role in the soil-plant system, mainly in the dynamics of P, N, S, and micronutrients (Ryan and Graham 2018; Hallama et al. 2019). However, the effects of SS-OMFs on soil quality and the microbial community are still unknown, especially in acidic soils (Togun and Akanbi 2013; Samuel et al. 2017; Lopes et al. 2018). The mixture of organic residues such as SS with mineral sources can cause beneficial effects in some microbial groups, improving soil quality, but deleterious in others, which can influence the availability of nutrients for plants and crop productivity (Chen et al. 2016).

The use of SS-OMFs will allow the use of this residue in tropical soils since it will be possible to explore its potentialities such as the presence of micronutrients and OM, while it promotes the reduction of dependence on MFs and the costs with the implantation of new landfills. Understanding the behavior of the different physical forms and their residual effect on the soil, as well as the possibility of using a lower rate of application compared to MF, can optimize nutrient cycling, and reduce environmental impacts generated by the exclusive application of SS in the environment. In this sense, it is essential to understand the soil quality, since studies focused on Brazilian conditions are scarce. We expected that this holistic approach will make it possible to direct management strategies for rural producers in the search for sustainable tropical agroecosystems.

1.1 General Objectives and Hypotheses

This thesis aimed to propose, synthesize and characterize an organomineral fertilizer from raw sewage sludge (SS-OMF) with a history of environmental restrictions due to heavy metal contents, and to explore the effects of its application in a no-tillage system of tropical cultivation (soybean - maize off-season succession). We hypothesized that: i) SS-OMF is an adequate and environmentally safe source of micronutrients for plants, mainly zinc; ii) the slow release of nutrients from the SS-OMF promotes better use of P than mineral fertilization, resulting in higher productivity; iii) the physical forms of presentation of the SS-OMF influences the availability of zinc and heavy metals in the soil and the plant; iv) lower application rate of SS-OMF (70% of P_2O_5 soybean needs) promotes the similar productivity as mineral fertilizer (100% of P_2O_5 soybean needs), and; v) the better agronomic performance of plants that receive SS-OMF compared to mineral fertilization is associated with improvements in bioindicators of soil quality in the rhizosphere of soybeans and maize off-season plants.

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2. SEWAGE SLUDGE IN THE MANUFACTURE OF ORGANOMINERAL FERTILIZER: PARADIGMA CHANGE TOWARDS SUSTAINABLE AGRICULTURE. A BIBLIOMETRIC LITERATURE REVIEW

Abstract

Sewage sludge (SS) is a by-product generated in wastewater treatment plants and its use in agriculture is advantageous since it has organic C and nutrients. However, the possibility of environmental contamination, mainly by heavy metals, it's a concern. The synthesis of organomineral fertilizers using SS (SS-OMFs) has been presented as a safer strategy to dispose of SS in agriculture since contaminants are diluted during fertilizer manufacture. Despite the importance of the subject, no critical review based on bibliometric data has yet been carried out. The VOSviewer software was used to perform cluster analysis using the Web of Knowledge database between 2012-2023 to identify the current research focused on the use of SS-OMFs, their potentialities, and challenges. Long-term research under realistic field conditions is needed to ratify the agronomic efficacy, residual effects, and economic feasibility of synthesizing of SS-OMFs to encourage investments. The present review summarizes the current literature and will help to direct future research on the use of SS-OMFs and their effects in agroecosystems.

Keywords: Biosolid, Bibliometric analysis, Sustainability, Biofertilizer, Waste management, VOSviewer.

2.1 Introduction

Proper management of organic wastes, such as sewage sludge (SS), is one of the main bottlenecks in building a sustainable society. SS is a semi-solid by-product generated in wastewater treatment plants (WWTPs), consisting of a mixture of organic (proteins, carbohydrates, fats, cellulose, etc.), inorganic contaminants such as Ni, Zn, Hg, Cu, and Fe microorganisms, potentially toxic substances (drugs, persistent organic pollutants, microplastics, etc.) and water (80-90% humidity) (Cieślik et al., 2015; Raheem et al., 2018; Khanh Nguyen et al., 2021). About 13 million tons (dry basis) of SS have generated annually in the world, landfills are its most popular destination (~40%) (Grobelak et al., 2019; Hu et al., 2022), and its processing represents about half of the operational costs of the WWTPs (Mateo-Sagasta et al., 2015). Therefore, a paradigm change is needed to face the rapid population growth (8 billion people in 2022): SS should not be looked at as a reject or waste, but as an organic residue with potential for agricultural use, promoting nutrient recycling and carbon sequestration in arable soils. For this, efficient and economic strategies are needed to ensure a safe SS destination.

SS presents considerable amounts of nutrients for plants, mainly N, P, Zn, and Ni, besides organic carbon (C) (Collivignarelli et al., 2020), turning it into an interesting organic matrix in the manufacture of organomineral fertilizers (OMFs) and thus favoring its safe use in agriculture (Antille et al., 2017; Kominko et al., 2019). As its name suggests, OMFs comprehend the physical mixture of mineral fertilizers, such as KCl (potassium chloride), MAP (monoammonium phosphate), and DAP (monoammonium phosphate), with organic residues, such as animal manure, poultry litter, food wastes, and SS (Smith et al., 2020). The direct application of SS to agricultural soils is often associated with environmental contamination issues, mainly related to the presence of high contents of hazardous trace elements in soils (Sakurada et al., 2016). However, the use of SS as the organic matrix in the synthesis of OMFs seems to be a promising sustainable strategy for its safe use in agriculture, since it is applied at much lower rates than applying strictly SS, the contents of hazardous trace elements in the OMF are diluted during formulating, its transport becomes economically feasible, and its application in the field is facilitated (Kominko et al., 2017; Seiple et al., 2020). OMFs can also improve the sorption and complexation of hazardous trace elements and pesticides, soil aggregation, and water flow, as well as reduce fertilizer saline effects and nutrient leaching, improving the soil quality (Lynch 2015; Audu and Samuel 2015; Geng et al., 2019).

Here, a bibliometric analysis was performed regarding the use and manufacture of organomineral fertilizers based on sewage sludge (SS-OMFs) to provide the main hotspots and gaps on the subject as well as to guide future research needs, supporting public policies and promoting advances in the search for agricultural sustainability. Our research was carried out using the Web of Knowledge database and the last years of publications (2012 - 2023), using the free-VOSviewer software as a tool to identify the main authors, countries, documents, keywords, and citations.

2.2 Material and methods

2.2.1 Database and search criteria

The database used was the Web of Knowledge (WOK), widely used in the construction of bibliometric analyzes focused on the Earth sciences due to its quality and multidisciplinary approach (Alryalat et al. 2019). To understand the research landscape associated with the synthesis and agronomic use of organomineral fertilizers (OMF), regardless of the organic matrix, the script search used was: "organomineral fertili?er*" OR "organo-mineral fertili?er" OR “organic mineral fertili?er*" OR “bio based fertili?er*" (topics). To direct the use of OMFs from sewage sludge (SS-OMFs), the Boolean operator AND was used and the complete search script consisted of: “organomineral fertili?er*” OR “ organo-mineral fertilizer*” OR “organic mineral fertili?er*" OR “bio based fertili?er*" AND “sewage sludge*” OR “biosolid*”. In both approaches, the time interval was 2012-01-01 until 2023-06-01. All types of publications were considered (original articles, reviews, conference articles, and early access). To ensure the quality of the dataset, data pre-processing was performed removing duplicate information in the original file with the aid of Microsoft Excel software.

2.2.2 Bibliometric analysis

The tool used for bibliometric analysis was the free software VOSviewer, version 1.6.19 (van Eck and Waltman., 2010). The analyses performed were a) C**o-occurrence** of keywords, considering “authors keyword” which allows maintaining the keywords assigned by the authors themselves, excluding those indexed by the database, considering the minimum number of occurrences equal to one; b) **Citation** by *i*) documents; *ii*) authors (minimum number of documents of an author equal to one and minimum number of citations of an author equal to zero. In all analyses, the counting method selected was “full counting”.

2.3. The panoramic view of the research on SS-OMFs

In the last years, 346 documents were published on OMFs in the Web of Knowledge database, regardless of the organic matrix. The interest in SS-OMFs is more recent, totalizing 67 published documents (Fig. 1A), distributed mainly in the areas of Environmental Sciences, Agronomy, and Engineering Environmental (Fig. 1B), evidencing an environmental and agronomic overlap in this topic, representing 47% of publications. The increase in food prices in the world due to fluctuations in the oil market encouraged the search for alternative sources of nutrients to meet the demands of agricultural systems, highly dependent on mineral inputs (Peersman et al., 2021). In addition, more restrictive legislation regarding SS disposal in sanitary landfills came into force in several countries, mainly in the European Union, increasing the pressure to safely direct SS to crops’ nutrition (Kominko et al., 2017)

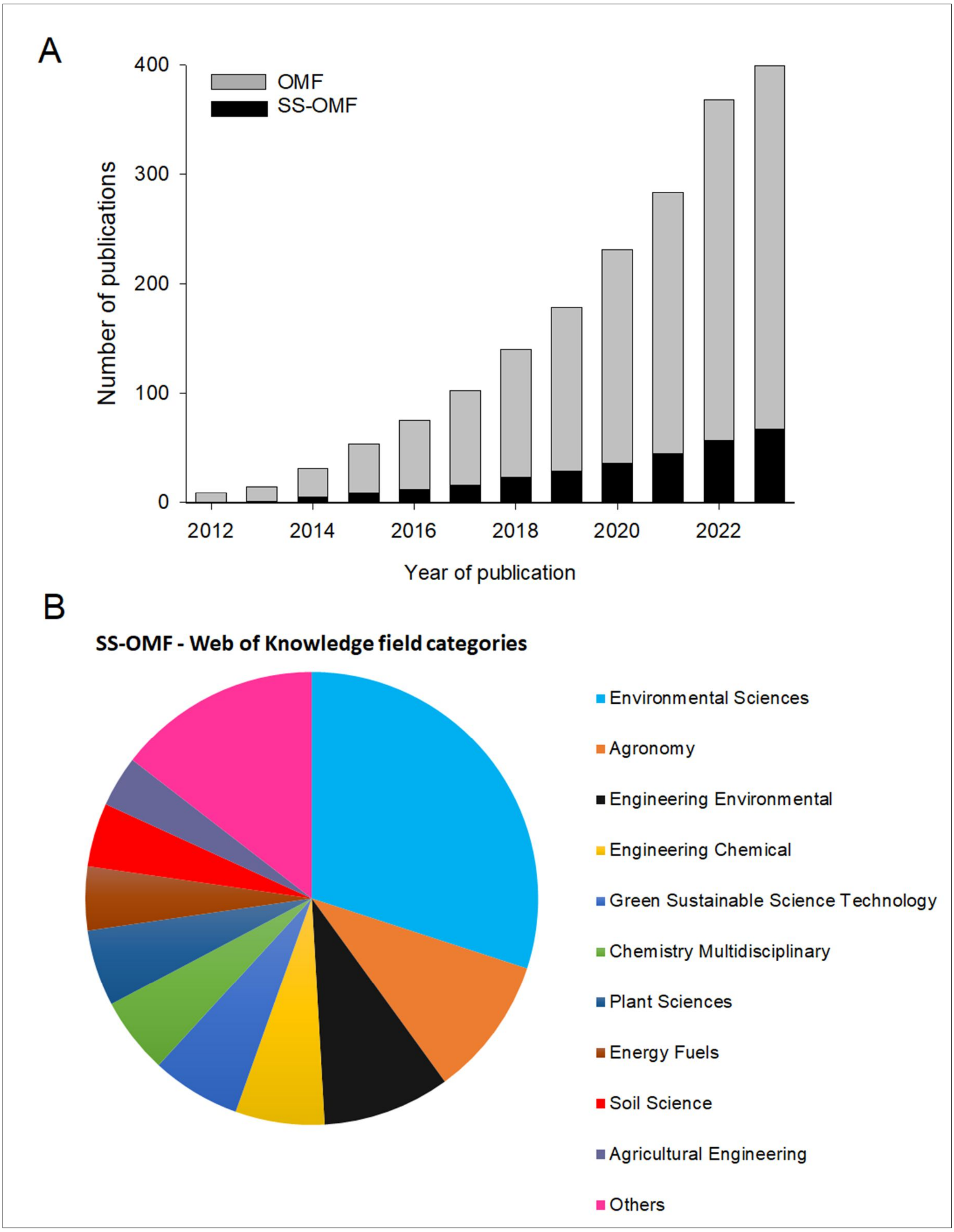

Fig. 1. Overview of SS-OMFs (organomineral fertilizer from sewage sludge) documents published between 2012-2023. A) Accumulated number of documents related to OMF (organomineral fertilizer) and SS-OMF. B) SS-OMFs published documents distributed in Web of Knowledge categories.

2.4 Most cited publications

The number of citations was regarded to select the most relevant works, cited authors, and organizations (Fig. 2). The most cited documents and authors reveal the Poland's relevance in this field of study. The review by Chojnacka et al (2020), associated with Wroclaw University of Science and Technology, Poland, appears as the first most cited document (174) entitled "*Bio-based fertilizers: A practical approach towards circular economy*" published in the "*Bioresource Technology"* (orange cluster) (Fig. 2). The valorization of various wastes such as chicken manure, sugarcane straw, and manures are explored, including SS, in the form of ash, composted, supplemented with mineral sources of nutrients and mixed with other wastes. These materials are an alternative to the circular economy in the fertilizer market, which is dependent on non-renewable sources of nutrients. For example, it is estimated that P deposits have a useful life of between 50-400 years.

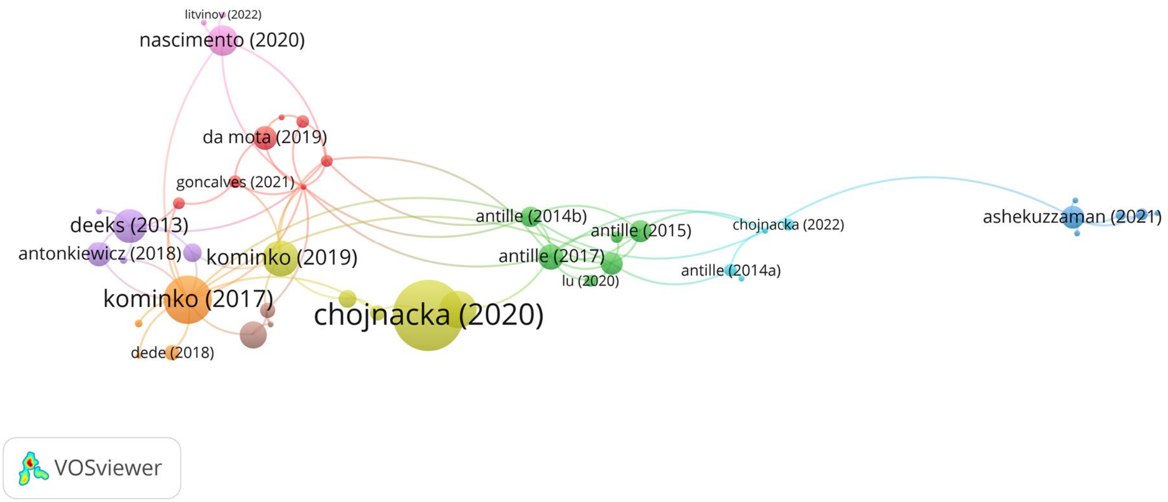

Fig. 2. Map of network visualization of analyses of citation by documents. The size of the nodes refers to the number of links per document and the lines represent the strength of a link between documents.

The review by Kominko et al (2017) is the second most cited document (80) (orange cluster), entitled *"The Possibility of organo-mineral fertilizer production from sewage sludge"* and published in the *"Waste and Biomass Valorization"* journal, associated with Cracow University of Technology, Poland (Fig. 2). It is the first work to discuss the state of the art on the synthesis and application of SS-OMFs and raising concerns to the needs of seeking local solutions for proper SS disposal due to differences in their physical and chemical attributes since sewage collection and their treatments differ within the WWTP. In addition, SS-OMFs were presented as a viable alternative to safely supply nutrients to plants, avoiding soil contamination by hazardous trace elements when compared to strictly using SS; and SS-OMFs were as efficient as mineral fertilizers in terms of crop yields, having the advantages of slowly releasing nutrients to plants and inputting organic-C to soils. They also reported that granulation or pelletization of the SS-OMFs is advantageous since they facilitate transport, storage, and field application.

The third most cited article in this field (45) is entitled *"Potentiality of sewage sludge-based organo-mineral fertilizer production in Poland considering nutrient value, heavy metal content and phytotoxicity for rapeseed crops"*, which was published in the "*Journal of Environmental Management*" by Kominko et al. (2019) (yellow cluster) (Fig. 2). An SS-poultry litter-OMF granulated with mineral acids was synthesized and its application increased rapeseed stalk development but suppressed root development, but heavy metal contents were below the threshold values settled by Poland legislation. In addition, it allowed the recycling of 82-140 tons of P_2O_5 and 42-73 tons of N from the WWTP.

Deeks et al (2013) the fourth place in the ranking of most cited documents (40) (purple cluster), entitled *"A new sludge-derived organo-mineral fertilizer gives similar crop yields as conventional fertilizers"* and published at the *"Agronomy for Sustainable Development"* journal (Fig. 2). This article addressed the synthesis of an SS-OMF and its agronomic efficacy in the development of wheat, oilseed rape, barley, beans, and forage maize. The SS-OMF application did not significantly differ from the mineral fertilization (ammonium nitrate plus muriate of potash) and the heavy metal contents in the soil did not exceed the threshold values set by the local legislation.

The most cited Brazilian article that effectively evaluates the agricultural use of SS-OMF (20) is entitled *"Biosolid and sugarcane filter cake in the composition of organomineral fertilizer on soybean responses"* published in the *"International Journal of Recycling of Organic Waste in Agriculture"* (Mota et al., 2019). The SS-OMF and the sugarcane filter cake-OMF, both in pelletized forms, promoted greater soybean heights than the conventional mineral fertilization, and soybean stem diameter was also greater when 75% of the recommended fertilizer rate was used at a greenhouse trial and 90 days after planting. Compared to strict mineral fertilization, SS-OMFs only showed greater agronomic efficiency in the studies developed under tropical conditions. It may suggest that these fertilizers should have higher efficiency in the use of nutrients in tropical soils, in which weathering tends to be more intense, as well as the degradation of organic matter and the leaching of nutrients, turning the soil more acids; and therefore, the addition of organic material should be more advantageous (Scopel et al., 2013).

2.5 Analysis of keywords

The keywords co-occurrence mapping may help to identify the main themes or hotspots on the research topic (Fig. 3) (van Eck and Waltman., 2010). The most representative ones tend to be in the center of the map, with bigger nodes and thick lines, while the fewer usual ones tend to be shown in the peripheral area, with smaller icons and thinner lines between the nodes (Fig. 3A). The initial investigation resulted in 119 keywords, but the three major ones were: “organomineral fertilizer” (14 occurrences), “sewage sludge” (13 occurrences), and “heavy metal” (7 occurrences), indicating a clear concern with toxicity caused by heavy metals when SS is directed to soils (Fig. 3B). However, the use of SS-OMFs should minimize possible problems with heavy metals when compared to using sewage sludge, since the concentration of nutrients reduces the loads of heavy metals in the soils, adding value and increasing the radius of distribution.

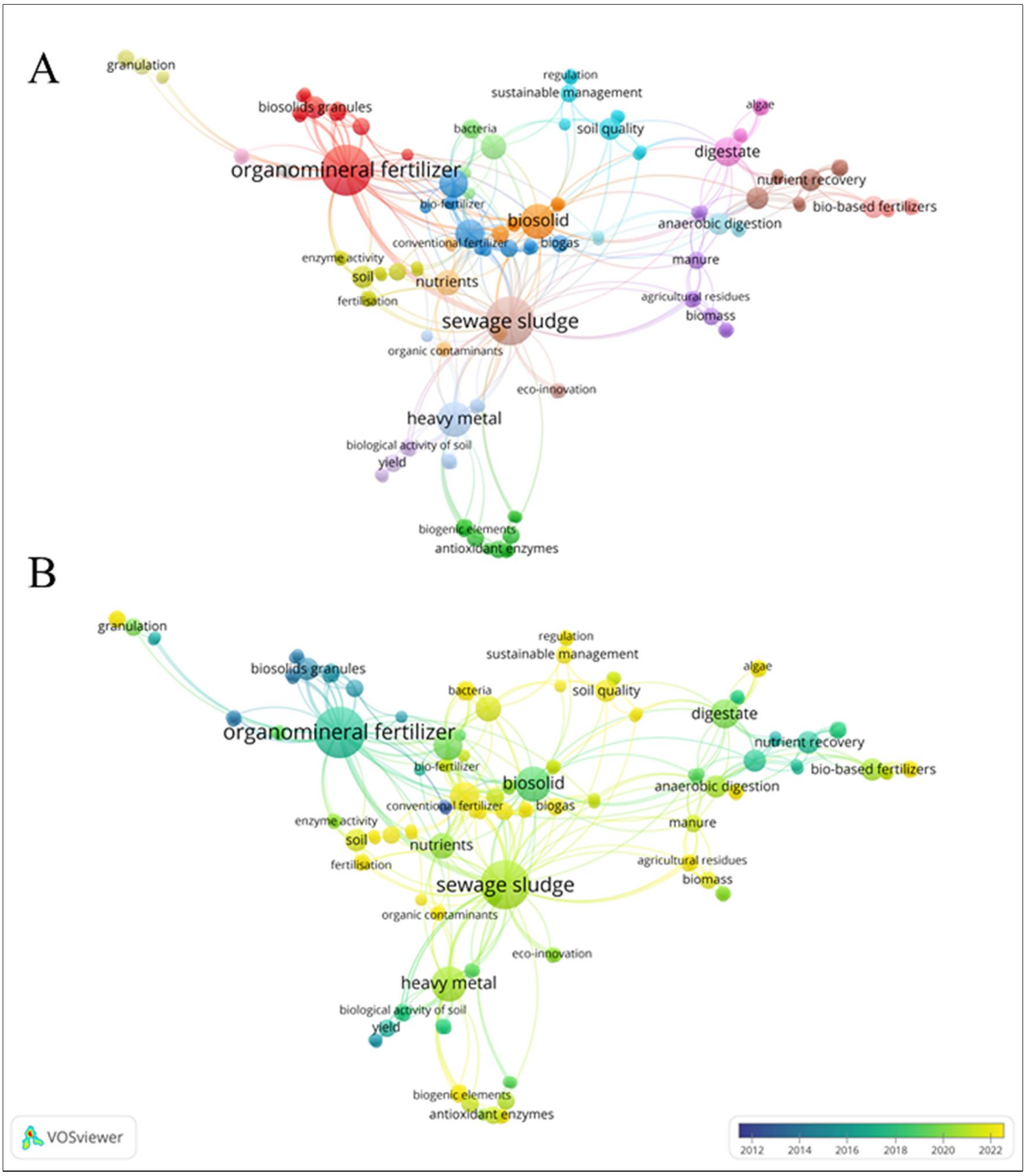

Fig. 3. Keyword co-occurrence mapping. A) Network visualization. B) Overlay visualization. The lines represent the strongest links between keywords and different colors represent clusters.

The main clusters formed by the author's keywords were (Fig. 3A): *i*) red cluster (organomineral fertilizer, biosolids granules, granulation, and N availability), concerned mainly with evaluating the efficacy of the different SS-OMF physical forms, being the granule form the most relevant; *ii*) blue cluster (heavy metal, pollution assessment, and phytoremediation), concerned mainly with environmental aspects associated with SS-OMF application; *iii*) navy blue cluster (circular economy, phosphorus, bio-fertilizer) concerned reuse of nutrients mainly

P and N, and use of SS as biogas; *iv*) light blue cluster (soil quality, tropical soil, sustainable management, and environmental impacts) concerned with the biological quality of the soil and environmental safety, and; v) light brown cluster the keyword “sewage sludge”, which forms the highest number of connections. Keywords related to “long-term” studies were not observed (Fig. 3A), showing that the use of SS-OMFs in agriculture still has a vast field to be explored due to the lack of long-term studies performed under more realistic field conditions, allowing to evaluate of its agronomic efficacy as well as environmental concerns, such as the possibility of hazardous trace elements and microplastics accumulation and the development of antibiotics resistant genes. These concerns are inherent to the composition of the SS, but the manufacture SS-OMFs should lessen them.

Looking at the timeline (Fig. 3B), the terms “organomineral fertilizer” and “sewage sludge” started to be connected only in works published from 2016 onwards, which corroborates that the interest in using SS-OMFs in agriculture is recent and mainly related to the need for proper disposal of the SS. Aspects related to the biological quality of the soil, such as the keywords “soil quality”, “bacteria” and “sustainable management” started to be explored in the last 2 years (yellow) and are hotspots in this field of study (Fig. 3B). The heavy metal and organic contaminants contents hold up in recent publications, which shows that a consensus on the environmental safety of using SS-based fertilizers is far from being reached (Fig. 3B).

2.6 Use of SS-OMFs in agriculture

2.6.1 Formulation and manufacture

The formulation of SS-OMFs allows for adjustment of the needs of nutrients to crops and soil attributes. The supplementation with potassium (K) should not be overlooked due to its low content in the SS (Grobelak et al., 2019). K has a high solubility in water, and it remains in the liquid phase during sewage treatment processes. The proportion of SS in the formulation ranges from nearly 30 to 60% and must comply with the legislation of each country. In Brazil (IN 61/2020), solid SS-OMFs should have at least organic C content of 8% and CEC of 80 $\text{mmol}_c\ \text{kg}^{-1}$, and at most 20% of moisture. In Europe (EU 2019/1009), solid SS-OMFs should have at least 7.5% of organic C content.

The manufacture of SS-OMFs is a relatively simple and easily reproducible process, comprehending: *i*) SS is collected in WWTPs often having moisture around 70 to 80%, which can be added with hydrated lime (Gonçalves et al., 2020; Hawrot-Paw et al., 2022) or not (Rodrigues et al., 2021); *ii*) SS can be used compostable (Kominko et al., 2019; Silva et al.,

2020) or non-compostable (Rodrigues et al., 2021; Deeks et al., 2013); *iii*) SS is dried in the sun or an oven with forced air circulation (65 to 80 °C), until the moisture of 20 to 30% (Rodrigues et al., 2021; Deeks et al., 2013); *iv*) then, SS is supplemented with mineral fertilizers, preferably, mineral sources with the highest concentrations of nutrients. To produce granules or pellets, both SS and mineral fertilizers should be crushed and sieved.

The granulation of the SS-OMFs involves the use of a granulator mixer for granulation on a laboratory scale, for example, for SS-poultry litter-OMFs using a liquid feeder and granule classifier, using a disk granulation with 80 cm, inclination of 35°, and rotation of 60 rpm (Kominko et al., 2019). Into the mixture, a dilute solution of nitric and sulfuric acid was added, and the granules were dried at 70 °C for 3 hours, which is essential to prevent the proliferation of fungi (Kasprzycka et al. 2016; Kominko et al., 2019). This approach allows the recycling of 82-140 t of P_2O_5 and 42-73 t of N annually in WWTPs in Poland (Kominko et al., 2019). Another possibility is to proceed with granulation in a tumbling evaporator, obtaining granules of 3-6 mm (Deeks et al., 2013). There is evidence that the ideal SS-OMF granule size (25% dry solids) should be 1.1-5.5 mm, with 80% between 2.25-4.4 mm for proper distribution (Antille et al., 2013). Another example is patent PL233754, which consists of the production of adequate granulated SS-OMF with 2-6 mm in diameter, from stabilized SS, with the addition of lime, gypsum, ammonium carbonate, dolomite, and microcrystalline cellulose (Glodniok et al., 2021).

The pelletization of SS-OMFs is another interesting way of presenting this product, in which the initial investment for the manufacturing process is usually lower than for granulation (Runge et al., 2018) but the production cost is higher since requires the use of a high-pressure pelletizing matrix (Rodrigues et al., 2021). However, pelletization might be advantageous since it seems to release the nutrients more slowly, avoiding the leaching of N and K mainly (Antille et al., 2013). The pellet sizes in the different studies ranged from 3.9 mm in diameter and 9.1 mm in length (Gonçalves et al., 2021) to 3 mm in diameter and 10 mm in length (Rodrigues et al., 2021). In both processes, it is important to add a binding agent to the formulation, such as acids, bentonite, or pregelatinized starch.

Here, one of the main challenges is the heterogeneity of the SS that can affect the reproducibility of the final product and improve the quality control of both physical and chemical properties of the products (nutrient contents, particle size, and granulometric distribution) (Antonkiewicz et al., 2018). However, due to operational ease, the incorporation of the synthesis of SS-OMFs has the potential to reduce the environmental impacts of sanitation systems in sub-Saharan countries (Agunyo et al., 2019).

2.6.2 Major agricultural findings

Agronomic implications of the use of OMFs are still little known, since soil responses, *i.e.*, changes in pH, CEC, and salinity, are highly dependent on the organic matrix used (Bouhia et al., 2022). Most of the agricultural studies with SS-OMFs were short-term (1-3 years experiment) and conducted in greenhouse conditions (Table 1). This is an essential aspect, since most work claims that the use of SS-OMF can replace mineral fertilizers, however, no results obtained in the literature effectively support this statement (Table 1). Despite the use of SS-OMF promoting similar responses to mineral fertilization in vegetative development, such as shoot and root dry mass, height, and stem diameter, will not necessarily reflect in equivalent productivity (Antille et al., 2014; da Mota et al., 2019; Silva et al., 2020).

It is widespread that the use of SS-OMFs increases the efficiency of nutrients such as N, P, and K by plants due to its slow release, however, the main agronomic findings are not conclusive (Table 1). Most N slow release occurs about 30 days after soil application (40-70%), which favors the reduction of the loss of this nutrient in the environment, however, it may not be suitable for crops that need N available in the early stages of development (Deeks et al., 2013; Antille et al., 2014).

For tropical regions, where P is highly adsorbed on Fe, Mn, and Al oxyhydroxides in soil, it is speculated that the use of organic matter present in SS-OMFs may act as a P adsorption site, favoring the access of these elements through the roots of plants (Kominko et al., 2017; Crusciol et al., 2020; Netto-Ferreira et al., 2023). The organomineral formulation guarantees the soluble levels of P, however, it is worth considering that there is an organic and inorganic fraction from SS that is not readily available for plant uptake. Most of the P in SS is in inorganic form (~70% anaerobically digested sludge) and approximately 87% in the form of non-apatite inorganic phosphorus fraction ($AlPO_4$ and $FePO_4$), with low solubility (Nosek et al., 2020; Chen et al., 2021). However, these aspects are not explored by the literature on SS-OMFs (Table 1).

The use of SS-OMFs may promote secondary benefits in crop performance compared to mineral sources associated with the organic compound, beyond the nutrient input. Its use as an inoculant carrier for *Azospirillum brasilienses* in soybean plants proved to be an efficient environment for seed treatment, due to the presence of humic substances from SS that Interact with plant auxin synthesis, cellular respiration, and protein synthesis, promoting a reduction in the activity of physiological stress parameters such as superoxide dismutase, lipid peroxidation and hydrogen peroxide concentration (de Barros et al., 2022). Furthermore, germination tests show that, at adequate rates of application, the use of SS-OMF has a stimulating effect on the hypocotyl of watercress, sorghum, and mustard, not suppressing plant germination (Kominko et al., 2021). The same benefits were observed with the use of liquid SS-OMF in the germination and development of pea and barley plants due to the presence of biologically active substances such as organics, vitamins, and amino acids (Ivanchenko et al., 2021).

The effect of SS-OMFs on the biological quality of the soil is a recent approach and an important way to verify their possible negative or positive impacts in agroecosystems (Table 1) (Rodrigues et al., 2021; Dindar, 2020). It is known that the use of bio-base fertilizers such as SS-OMFs contributes to the maintenance of soil quality, which is fundamental in controlling phytopathogens, stimulating plant growth and resistance to attacks by pests and diseases, which is reflected in the crop yield (Buneman et al., 2018; Jing et al., 2022; Bhattacharya et al., 2023). A study in a greenhouse showed little effect on the physiology of corn fertilized with SS-OMF. However, the microbial activity (CO_2 emission) and the number of bacteria of the genus *Bacillus* were greater in the soil that received this fertilizer instead of mineral fertilizers (Hawrot-Paw et al. 2022). This bacterial genus plays an important role in the health and growth of plants, performing multiple ecological functions in the soil, from nutrient cycling and phytohormone production to tolerance to biotic and abiotic stresses in plants (Saxena et al. 2020).

Table 1. Studies with organomineral fertilizers having sewage sludge as the major organic matrix (SS-OMF) from journals with either impact factor (IF) >1 or citation number (CN) ≥ 5, developed under greenhouse or field conditions. Their major findings and conclusions (continue)

Article	IF	CN	Greenhouse
Antille et al., 2014	1.58	6	**Findings:** N release from an SS-OMF occurred mainly within 30 days following soil application accounting for 40-70% of the total N applied as fertilizer, with further release of 10-30% in the following 30-60 days. Compared to urea, these values were 17% to 26% higher. **Conclusions:** The timing of OMF application requires investigation at the field-scale to optimize nutrient use efficiency and reduce the risk of nutrient losses to the environment.
Antille et al., 2014	1.58	14	**Findings:** When applied at the same rates but SS was treated with Fe, the resulting SS-OMF (15-4-4) presented much lower (< 6.5% of applied P) P availability than single superphosphate (0-18-0) [16% (sandy loam) to 46% (clay), depending on soil texture] after 90 days of incubation. **Conclusions:** The timing of OMF application requires investigation at the field-scale to optimize nutrient use efficiency and reduce the risk of nutrient losses to the environment.
Antille et al., 2014	1.58	17	**Findings:** After 3 years of continuous applications, SS-OMFs (15-4-4 and 10-4-4) increased the dry mass yield of ryegrass by 2-27% compared with biosolids but to a lesser extent than urea (range: 17-55%). Urea and SS-OMFs showed significantly greater N recoveries than biosolids. Soil extractable P levels remained close to constant after 3 years of continuous SS-OMF applications but increased with biosolids and decreased with urea applications. **Conclusions:** Both SS-OMF formulations are suitable for application in ryegrass. They seem to replenish P off-take by grass crops thereby maintaining soil P levels overtime.
da Mota et al., 2019	2.92	20	**Findings:** Compared to mineral fertilization, SS-OMF at rates of P_2O_5 > 50% increased soybean steam diameter and plant height but did not affect chlorophyll's content at 30 days after soybean. **Conclusion:** OMF based on sewage sludge and sugarcane filter cake can be used to replace mineral fertilization for soybean crop production.
Silva et al., 2020	2.11	6	**Findings:** SS-OMF at P_2O_5 rate equal to 100% increased root dry mass of soybeans at 30 days after sowing, whereas rates > 50% increased stem and leaf dry masses at 80 days after sowing, but no differences were observed in plant antioxidant enzymes, except for catalases. **Conclusions:** The use of biofertilizers based on sewage sludge provides a 25% reduction in the required dose of mineral fertilizer, leading to a greater growth increase.

Table 1. Studies with organomineral fertilizers having sewage sludge as the major organic matrix (SS-OMF) from journals with either impact factor (IF) >1 or citation number (CN) ≥ 5, developed under greenhouse or field conditions. Their major findings and conclusions (to be continued)

Rodrigues et al., 2021	11.07	5	**Findings:** Regardless of its physical form (powder, granule, or pellet) and compared to mineral fertilization, SS-OMFs (4-8-8) enhanced nutrients accumulation (mainly N, P, and B in the CLY soil), pod numbers, and nodulation, as well as soil microbial activity of the soils. The studied SS-OMFs worked as sources of Zn and B to plants. **Conclusions:** The use of SS-OMFs should result in higher soybean yields as well as open new possibilities in the search for an economically viable and environmentally safe strategy for SS disposal. Further research is needed to assess their application rates, residual effects, and economic viability under more realistic field conditions.
Barros et al., 2022	2.92	1	**Findings:** Inoculation of SS-OMF with *Azospirillum brasiliensis* changed the activities of superoxide dismutase, lipid peroxidation, and hydrogen peroxide inoculation compared to the control (without inoculants and fertilizers), whereas co-inoculation of *A. brasiliensis* with *Bradyrhizobium japonicum* enhanced proline activity. **Conclusions:** SS-OMF is an efficient carrier of *A. brasiliensis* for the inoculation of soybean seeds, improving plants' protection against stresses in critical stages of soybean development.
Rodrigues et al., 2023	3.87	0	**Findings:** In the short term (60 days), the SS-OMF physical forms (powder, granule, and pellet) did not affect soybean vegetative development, but higher application rates increased K and P accumulation as well as its shoot dry mass compared to mineral fertilization, but decreased root dry mass and nodulation. In the clay soil, more P and K were accumulated in soybeans when supplied via SS-OMFs. **Conclusions:** Overall, 70% of P full rate for the SS-OMFs would be recommended for clay soils whereas 100% would be needed for sandier soils. SS-OMF manufacture is a safe strategy for warrantying nutrient and organic matter recycles in tropical regions that are highly dependent on the importation of mineral fertilizers.
Kominko et al., 2021	8.91	18	**Findings:** There was an increase in the fresh biomass oh the aerial part with the use of SS-OMF for rapeseed (75-138%), maize (96–138%), and sunflower (23–54%) compared to control without fertilization, however, the use of SS-OMF doses above the recommended inhibited the germination and root growth of cress, sorghum, and mustard in a germination test. **Conclusions:** SS-OMF is a promising alternative for fertilization of rape, maize, and sunflower in adequate rates.

Table 1. Studies with organomineral fertilizers having sewage sludge as the major organic matrix (SS-OMF) from journals with either impact factor (IF) >1 or citation number (CN) ≥ 5, developed under greenhouse or field conditions. Their major findings and conclusions (to be continued)

(Netto-Ferreira et al. 2023)	4.89	0	**Findings:** SS-OMFs increased the P and K efficiency index compared to biosolids (with and without granulation). SS-OMFs did not register N immobilization, but they were effective in decreasing P immobilization in the soil when compared to a single P source. Conclusions: Granulated SS-OMFs increase the efficiency and availability of nutrients, but the presence of more than one macronutrient affects nutrient release dynamics. SS-OMFs have the potential to increase nutrient use efficiency and decrease agriculture's reliance on mineral fertilizers.
			Field
(Deeks et al. 2013)	7.83	40	**Findings:** There was no significant difference in crop yields between mineral and SS-OMFs over three trial years, except for one crop (winter wheat yielded 20% when amended with SS-OMF). Soil fertility and N uptake efficiency were similar for both fertilizers (SS-OMF and mineral fertilizer). **Conclusions:** Despite its organic source, N uptake by the crops was efficient after applying SS-OMFs. This indicates improved soil fertility possibly associated with increased soil microbial activity that assists in the mineralization of nutrients from OMFs. The novelty of producing fertilizer from a renewable resource without compromising crop yield and supporting sustainable agriculture was a key feature of this work. It showed that is possible to convert waste into resources.
(Pawlett et al. 2015)	6.15	6	**Findings:** SS-OMFs (10-4-4 and 15-4-4) had similar ryegrass yields as the biosolid and urea applications individually after 3 years of consecutive application, but urea at a high rate (250 kg ha^{-1} of N) reduced yield in the first year (2011). Soil total carbon increased after biosolid and SS-OMF$_{10}$ applications. P uptake by plants was not affected by fertilizer sources. **Conclusions:** Comparable yields produced by the application of SS-OMFs compared to urea suggest that they are a promising alternative to inorganic fertilizers. In addition, changes in available P suggest that SS-OMFs replenished their uptake maintaining soil P levels. SS-OMFs can be utilized as a renewable source of fertilizer for ryegrass even in a high P soil without compromising yield whilst maintaining the P level.

Table 1. Studies with organomineral fertilizers having sewage sludge as the major organic matrix (SS-OMF) from journals with either impact factor (IF) >1 or citation number (CN) ≥ 5, developed under greenhouse or field conditions. Their major findings and conclusions (conclusion).

(Antille et al. 2017)	2.65	22	**Findings:** Average winter wheat grain yields for SS-OMFs (10-4-4 and 15-4-4) were 25% higher than with biosolids granules, but 20% lower than with urea. Grain-N recoveries were 31% for biosolids, ~ 40% for SS-OMFs, and 52% for urea. SS-OMFs did not change soil extractable P and soil P Index, but extractable P increased in biosolids and decreased in urea-treated soils, respectively. **Conclusions:** Differences in grain yield and yield-to-N responses between treatments were due to relative patterns of N release from fertilizer applied to soil influencing N uptake, biomass accumulation, and partitioning. The agronomic performance of winter wheat treated with SS-OMF may be optimized by applying a straight N fertilizer source as the first (40-50%) and SS-OMF as the second fertilizer application (50-60%), respectively. SS-OMF replaced P absorbed by the crop whereas soil K-Index was maintained when wheat was grown but declined when grass was grown for 2 years, suggesting that additional application of K fertilizer may be required.
(de Moraes et al. 2020)	0.45	5	**Findings:** No differences were observed in productivity, number of tillers, stem diameter, and height of sugarcane plants between SS-OMF and mineral fertilizer. SS-OMF rates increased productivity, height, and stem diameter of the sugarcane in the soil with lower soil fertility, but not in the soil with high fertility. However, the use of bio-stimulants did not contribute to sugarcane yield in the low fertility scenario but enhanced yield in the high fertility scenario. **Conclusions:** SS-OMFs and bio-stimulants are viable alternatives for sustainable fertilization and nutrient cycling in soils cultivated with sugarcane. They can replace mineral fertilization in low and high-fertility environments.
(Gonçalves et al. 2020)	3.75	6	**Findings:** There were no significant differences in the sugarcane attributes [stalks weight per hectare (t ha^{-1}), sugarcane productivity (t ha^{-1}), quantity of sugar per hectare (TSH, t ha^{-1})], and physicochemical properties of the juice [pol (%), Brix (%), purity (%), and fiber (%)] between SS-OMFs and the exclusive mineral fertilization. The recommended SS-OMF rate to obtain maximum quantitative and qualitative sugarcane attributes was between 102-109% of the mineral fertilizer rate. **Conclusions:** Sewage sludge is a viable alternative for OMF composition and could improve economic returns and minimize negative environmental impacts to exclusive mineral fertilization in sugarcane cultivation systems.

2.7 The issue of heavy metals and soil contamination

Although heavy metal contents are diluted when the SS is converted into SS-OMFs, there is still no consensus on the real safety of using these compounds in agricultural soils, mainly when using SS of industrial origin, in the long term. Currently published studies have shown that levels of heavy metals in soil that received SS-OMFs did not exceed threshold values (Deeks et al., 2013; Antille et al., 2017). Compared to using SS, its SS-OMF did not influence the contents of Cd and Pb in the soil but decreased the contents of hexavalent chromium (Cr^{6+}) (65%) > Cr (50%) > Ni (45%) > Pb (42%) > Fe (40%) > Zn and Cu (36%) (Rodrigues et al., 2021). In plants, the use of an SS-OMF promoted adequate plant development but increased the accumulation of heavy metals, mainly Cu and Ni but at low pollution levels and they are micronutrients to plants (Kominko et al., 2022). Thus, applying SS-OMFs is much safer than applying SS even considering that most of these studies were short-termed conditions. There is a numerous study in the literature performed in the long run (> 20 years) showing that heavy metal accumulations in soils amended with SSs are still not conclusive (Page, 1974; Alvarenga et al., 2015; Cieślik et al., 2015). So, amending SS-OMFs should be less concerning, especially when their set threshold values are respected according to the legislation in place. Most countries have clear and rigorous legislation on this subject.

Despite heavy metal contents being the most limiting factors in the use of SS and converted products, the presence of other contaminants requires monitoring (Zawadzki and Głodniok., 2021). Persistent organic pollutants (POPs) still require investigation in these fertilizers (Zhang et al. 2023). These pollutants are recognized by the Stockholm Convention and consist of several molecules such as polychlorinated biphenyls (PCBS), polychlorinated biphenyls (PCBS), pesticides, among others, which accumulate in the trophic chain and are associated with various diseases in humans and animals (Alharbi et al. 2018). Countries such as USA, UK, Canada and Poland do not monitor POPs in SS due to their low concentration, based on historical monitoring. However, Brazil, China and some European countries such as France and Germany, establish acceptable limits in SS (Kominko et al. 2017). In Brazil, the legislation (IN 61/2020) that regulates the synthesis of OMFs does not include limits for these molecules in the SS-OMF, only in the soil after its application (CETESB, 2021). The concentration of POPs in SS depends on the type of treatment by WWTPs and tends to be lower after composting (Haynes et al. 2009; Rigby et al. 2021).

The organic fraction of SS may contain more recalcitrance organic pollutants, as well as present antimicrobial resistance genes and pharmacological residues, due to its origin (Wei et al. 2018). There is a presence of pharmaceuticals in water using an SS-OMF such as ibuprofen, diclofenac and carbamazepine, however, in smaller amounts compared to the use of SS (Styszko et al. 2022). The concentrations of these drugs in the soil can be reduced due to the presence of biochemical and physical processes and that, despite the low risk to ecosystems, their monitoring is necessary. However, this aspect is not explored in works with SS-OMFs.

2.8. Conclusion

The use of organomineral fertilizers from sewage sludge (SS-OMF) has shown to be agronomically efficient and a relatively safe form of using sewage sludge (SS) in agriculture. Research on this topic is growing, but concentrated in the last five years and more studies performed under more realistic field conditions and in the long-term are needed to ratify their agronomic efficacy and residual effects, as well their environmental safety. The synthesis of SS-OMF decreases the concentration of heavy metals and can raise levels in plants, however, below levels considered dangerous for human health. Their use in tropical and developing countries, which often have a heavy deficiency of nutrients and dependence on imported mineral fertilizers, could be essential to warranty food to their population and proper use of the SS until new and economically viable strategies are into place, such as SS incineration. Unfortunately, the literature still lacks information on the economic feasibility of using SS-OMFs, which hinders public and private investments in this type of product, as well as the adhesion by rural producers and the development of efficient public policies.

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3. SYNTHESIS OF AN ORGANOMINERAL FERTILIZER FROM SEWAGE SLUDGE FOR USE IN THE SOYBEAN MAIZE-OFF SEASON SYSTEM: AGRONOMIC EFFICIENCY, ENVIRONMENTAL SAFETY, AND RESIDUAL EFFECT.

Abstract

Brazil is one of the main food-producing countries in the world. One of the bottlenecks for the expansion of its agriculture is the high dependence on nutrient mineral fertilizers. On the other hand, the proper disposal of sewage sludge (SS) is one of the great challenges for the sustainability of modern society. The SS is a source of organic matter and nutrients for plants. However, the possible presence of inorganic contaminants can make its use in agriculture unfeasible. Here, the use of an organomineral fertilizers from SS (SS-OMF) was studied as a promising alternative for SS proper destination and to supply part of the demand for mineral fertilizers (MF), mainly in tropical soils, such as from Brazil. Our objectives were: *i)* to evaluate the effects of applying an SS-OMF (4-8-8) on physicochemical parameters of soil, bioindicators of soil quality, and the agronomic response of soybean (*Glycine Max* L.) and residual effects on maize off-season (*Zea mays* L.), in successive cultivation, in the no-tillage system; *ii)* to establish the best SS-OMF physical form and rate of application; and evaluate possible environmental risks concern SS-OMF application. For that, three physical forms (Pw, powder; G, granule, and Pt, pellets) and two P_2O_5-rates (70% and 100% of soybean needs) of SS-OMF were compared to MF (100% P_2O_5-rate) and Ct (no P-applied), in a field experiment. Results showed that in a clay soil with medium P content, on soybean plants, the effects of the use of SS-OMF on development, productivity, nutritional status, nutrient accumulation, and soil nutrient content were similar to MF. The active nodules with SS-OMF in G and Pt physical forms were about twice as much Pw, MF, and Ct. The Pt showed a greater agronomic efficiency concerning the MF in crops. For maize off-season the Pw showed the worst responses among physical forms whose lower performance may be due to higher Fe concentration. in grains, higher levels of Pb and Zn and lower levels of K in the soil and lower beta glucosidase enzyme activity compared to MF. In both crops, the heavy metal levels in the soil-plant system were considered much smaller than those considered harmful to human health and the environment, indicating that SS is a safe organic matrix for the synthesis of OMFs. The use of SS-OMF is a promising alternative to adequate SS disposal and can replace the use of MF in soybean, with the potential to provide positive residual effects on maize off-season.

Keywords: Biosolid, Organic waste, Nutrient recycling, Microbial activity, Environmental safety, Biofertilizer

3.1 Introduction

Brazil is one of the main producers and exporters of food in the world, being the first in the production of soybean (*Glycine Max* L.) (153 million t^{-1}), a crop that occupies about 43.4 million hectares, and third concerning maize (*Zea mays* L.) (125 million t^{-1}), spread over 21.5 million hectares (N. C. of S. Conab, 2023). The maize off-season or second corn crop currently corresponds to approximately 70% of the corn produced in Brazil (Colussi and Schnitkey, 2021). It is cultivated mainly in the central and south-central regions of the country, usually planted between January and February, and harvested in winter, between April and June. Fertilizers represent around 1/3 of production costs in both crops (Aprosoja, 2023; Gomes et al., 2019). The Cerrado is the biome that concentrates around 50% of the production of these two *commodities* and most soils in the area are highly weathered, showing high levels of Fe, Mn, and Al oxyhydroxides and, consequently, high P fixation. Moreover, there is also intense K and B leaching due to the predominance of 1:1 clay minerals and low soil organic matter (SOM) contents, with micronutrient deficiency, intensified using formulations containing only NPK (Maranguit et al., 2017). For example, in these areas, Zn deficiency is considered one of the most limiting factors for the development of crops, aggravated by the high doses of limestone and phosphate employed (Nogueira et al., 2019). More than 50% of P and 85% of K fertilizers used in Brazilian agriculture are imported, evidencing the country's high dependence on mineral sources, being one of the major obstacles to food production (Pavinato et al., 2020; Sipert et al., 2020).

Thus, in regions such as the Brazilian Cerrado, mainly composed of medium to low-fertility soils (Lemes et al., 2019; Soterroni et al., 2019), the use of organomineral fertilizers from sewage sludge (SS-OMFs) is a viable and eco-friendly alternative for agricultural production. SS-OMFs are defined as the result of mixing or combining simple mineral fertilizers (MFs) and sewage sludge (SS) (Smith et al., 2020). It is estimated that the country generates 372,000 metric tons (dry basis) of SS annually (Mateo-Sagasta et al., 2015). This amount tends to increase after the recent enactment of the Basic Sanitation Legal Framework (Brasil, 2020), wich increased the pressure for the universalization of basic sanitation and the generation of SS, which is the main form of disposal of this waste in Brazil is landfilling (Ghini et al., 2016).

The SS-OMFs can promote agronomic and environmental benefits related to physical-chemical processes in tropical soils. For example, low molecular weight organic acids and humic and fulvic acids (Wu et al., 2013; Yu et al., 2013) can decrease P immobilization in the soil due to competition for P adsorption sites, making it more available for plant uptake (Fink et al., 2016). The slow-release of nutrients (mainly N and P) throughout mineralization optimizes its use by plants (Barcelos et al., 2019; Magela et al., 2019). This enables equivalent or lower rates of SS-OMFs being able to promote the same agronomic gains as MFs (Frazão et al., 2018; Gonçalves et al., 2020). Studies using SS-OMFs in tropical soils have indicated that rates of 25% lower can be as efficient as the recommended rate of MF in crops such as soybeans and sugarcane (Mota et al., 2019; Silva et al., 2020). This makes this type of fertilizer interesting from an economic point of view since it reduces the need for mineral inputs and still allows productive gains equal to or greater than MFs.

The segment of special fertilizers is growing in Brazil and showed an increase of 65% in revenues in 2022 compared to 2021, mainly in the states of São Paulo, Minas Gerais, and Mato Grosso (Abisolo, 2023). In a hypothetical scenario, using all the organic waste generated by the sugar-alcohol, swine farming, cattle, and poultry sectors in Brazil in the form of OMFs, it would have the potential to supply about half of the national demand for NPK, representing about US$ 1.1 billion (Cruz et al., 2017). In practical terms, the formulation of SS-OMFs allows the adjustment of the application rate according to the nutritional needs of the crops and soil fertility status (Magela et al., 2019). Due to the higher concentration of nutrients compared to SS, application rates of SS-OMFs are lower, reducing the risks of environmental contamination (Antille et al., 2017; Kominko et al., 2017a). The SSs generated in metropolitan regions such as those located in the State of São Paulo, usually with high levels of Ni and Zn (Nascimento et al., 2020), could be directed to the synthesis of SS-OMFs (Rodrigues et al., 2021). In addition to reducing costs and environmental impacts associated with the installation and operation of landfills for SS disposal, the use of SS-OMFs in agriculture facilitates its transport over long distances and its application in the field (Antille et al., 2013; Kominko et al., 2017a; Smith et al., 2020).

As important as the rates, the physical forms of SS-OMFs influence their final price and, consequently, their attractiveness to rural producers (Fadare et al., 2010; Szulc et al., 2021). The most used physical forms are powder, granules, and pellets, the last two being the most expensive (Romano et al., 2014). However, it is still unclear whether the physical forms are a determinant factor affecting the release and use of nutrients by plants or the residual effect of OMFs in subsequent crops (Fachini et al., 2021), This understanding is also necessary when using SS with high trace elements content, since it is not clear whether elements bioavailability in the soil solution is influenced by the SS-OMF particle arrangement.

There are gaps regarding possible synergistic or inhibitory effects of the mixture between the organic fraction (SS) and the MFs on soil quality indicators, such as enzymatic activity, glomalin, and microbial biomass. To understand changes in soil quality, bioindicators have been used to compare different forms of agroecosystem management, as is the case with the use of SS-OMFs. These bioindicators describe organisms that perform key functions in the soil ecosystem, mainly in the dynamics of C, N, P, S, and micronutrients (Schloter et al., 2018). The enzymatic activity of the soil and the quantification of easily exchangeable glomalin (EEG) (associated with the presence of arbuscular mycorrhizal fungi), and microbial biomass carbon (MBC), are examples of sensitive indicators of changes in soil microbial behavior (Hallama et al., 2019; Ryan and Graham, 2018). In a humid tropical climate, Wu et al. (2020) observed an increase in the availability of P in the soil in areas of tea cultivation that received organic residue compared to those fertilized only with MFs. This effect was related to the increase in microbial activity and biomass of microorganisms involved in the cycling of P. However, the implications for the soil microbial community due to the use of OMFs are little known, especially in acidic soils (Magela et al., 2019; Samuel, 2017; Togun and Akanbi, 2013). Results obtained in temperate regions indicate that there are differences in the soil microbial community between the application of pure organic material compared to its application mixed with mineral sources (Scrase et al., 2020).

Here, we evaluate the effects of applying an SS-OMF (4-8-8) on physicochemical and biological soil attributes and the agronomic response of soybean followed by maize, in a no-tillage system. Bioindicators of soil quality selected were: the activity of the enzymes β-glucosidase (C cycle), acid phosphatase (P cycle), arylsulfatase (S cycle), quantification of easily exchangeable glomalin (EEG), and carbon of microbial biomass (CMB). To verify the environmental safety of SS-OMF application, the semi-total content of trace elements in soil and plants was determined. To establish the best SS-OMF physical form and rate of application, three physical forms (Pw, powder; G, granule, and Pt, pellets) and two P_2O_5 rates (70% and 100% of the soybean need) were compared to MF and Ct (no P_2O_5-applied), in a field experiment conducted in an area of Cerrado biome. We hypothesized that: *i)* SS-OMFs are more efficient and have a greater residual effect than the MFs, allowing the use of a lower application rate (70% of the MF, based on the P_2O_5 requirement) with similar soybean productivity; *ii*) SS-OMF in G and Pt physical forms release nutrients more slowly than Pw, promoting a higher residual effect on the soil with nutritional gains for the maize off-season; *iii*) the greater efficiency of SS-OMFs is due to the presence of the organic matrix, which favors the cycling of nutrients and the activity of beneficial microbes associated with soil quality in soybean and maize off-season crops; *iv*) applying SS-OMF is environmentally safer than applying SS due to the lower application rate of the former, thus reducing the chances of soil contamination with toxic trace elements; and *v*) the heavy metals naturally present in the SS-OMF can be a safe source of micronutrients for soybean and maize off-season, even in soils lacking in these nutrients.

3.2 Material and Methods

3.2.1 Collection and characterization of sewage sludge

The sewage sludge (SS) was taken from Rio Preto wastewater treatment plant (WWTP), São José do Rio Preto city (~ 430,000 inhabitants), State of São Paulo, Brazil (20° 49' 12" S - 49° 22' 44" O). The treatment process is Upflow Anaerobic Sludge Blanket reactors and Conventional Activated Sludges. About 900 kg of SS cake [mixed source (domestic + industrial), but mainly domestic] were collected. The SS was air-dried in a covered shed, dried in a forced circulation stove at 65 ° C for 48 h, and milled (< 2 mm) (Fig. 1). A subsample was collected and characterized according to: *i*) physicochemical parameters of pH (H_2O and $CaCl_2$ 0.01 mol L^{-1}), moisture (60–65°C), density, organic carbon (via combustion), total contents of N (Kjeldahl), P total (P_2O_5, spectrophotometric determination of molybdovanadophosphoric acid) and soluble (neutral ammonium citrate plus water), K (K_2O) and Na (flame photometry), and S (barium sulfate gravimetry), with subsequent calculations of the C/N ratio and CEC (Teixeira et al., 2017), total organic matter (OM, via combustion) (Alcarde, 2009) (Table 1); *ii*) Heavy metals content of As, B, Ba, Cd, Cu, Cr, Cr^{6+}, Fe, Hg, Mn, Mo, Ni, Pb, Se, and Zn were extracted by Microwave Assisted Acid Digestion (3051A method; SW-846) (USEPA, 2007) and determined using Inductively coupled plasma - optical emission spectrometry (ICP-OES) (method 6010 D-1) (USEPA, 2014). Hg content was extracted by Cold Vapor Atomic Fluorescence Spectrometry (Method 245.7) (USEPA, 2005). Cr^{6+} content was obtained by alkaline digestion (3060A method) (USEPA, 1996) and ICP-OES, and *iii*) pathogenic agents of *Escherichia coli* (g TS^{-1}) (method 503) (USEPA, 1993) following the requirement of Brazilian legislation (Brazil, 2020a).

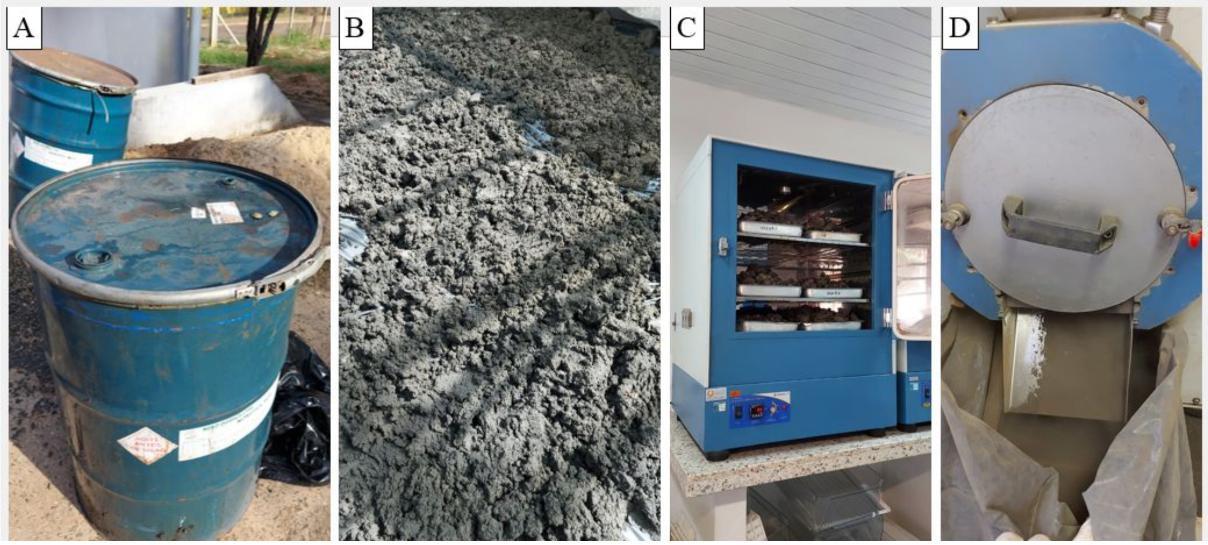

Fig 1. Drying stages of sewage sludge on canvas. forced air circulation oven. and grinding. A) Drum sealed with freshly collected sewage sludge; B) Sewage sludge in the process of drying in a greenhouse; C) Drying of sludge in a forced air circulation oven; D) Grinding sewage sludge to fine powder.

3.2.2 Synthesis of organomineral fertilizer and its physical forms

The organomineral fertilizer from sewage sludge (SS-OMF) formulation proposed was 4-8-8, intended for use in soybean cultivation. A previous study in a greenhouse indicated that the use of SS-OMF 4-8-8 promoted adequate development of the plants and did not cause suppressive effects on soybean nodulation, since the use of formulations containing N should be adjusted with caution (Rodrigues et al., 2021). The elaboration of the SS-OMF was carried out following legal requirements Normative Instruction 61 (Brazil, 2020b): *i)* organic carbon: minimum of 8%; *ii*) maximum humidity: 20%; and *iii*) minimum CEC: 80 mmolc kg^{-1}. From the analytical results of dry and ground SS, the SS-OMF was formulated and supplemented with simple mineral fertilizers, following the mass proportions (%): SS (58.5); monoammonium phosphate (MAP) (14.3); potassium chloride (KCl)

(13.7); dolomitic limestone (9.35); elementary sulfur (S^o) (1); boric acid (H_3BO_3) (0.15) and pregelatinized starch (3).

To obtain the mash mixture (Pw, powder), the mineral fertilizers MAP and KCl were ground in a knife mill (< 2 mm), and all the raw materials were weighed and mixed in a concrete mixer to homogenize the mixture. The Pw was used in the preparation of the granule (G) and pellet (Pt) physical forms. Two preliminary tests were carried out to obtain an adequate mass for the formation of the granules, being: a) granules formed with the use of raw materials without prior grinding; and b) granules formed using regrinding materials (a new grinding of dry and ground SS and dolomitic limestone, < 0.5 mm) (Appendix A). The granulation process was carried out by agglutination, using a high-intensity mixer-granulator, and the humidity of the mass was adjusted as necessary. The formed granules were dried in an oven with forced air circulation at 65°C until constant weight and classified in sieves ranging from 4.0 to 0.5 mm. The pelletizing process was carried out in a pellet machine, with 7.5 HP, and the mass moisture was adjusted to approximately 10%. The formed pellets were dried in air and in an oven with forced air circulation at 65°C until constant weight.

3.2.2.1 Physicochemical and structural characterization of SS-OMF physical forms

The same physicochemical parameters determined in the SS (item 3.2.1) were determined in the different physical forms of SS-OMF, with the inclusion of soluble K_2O in water (MAPA, 2017) To verify the presence of possible pathogenic microorganisms in the SS-OMF, tolerant coliforms, viable eggs of helminths and *Salmonella* sp were determined, as requested by official legislation for the use of organomineral fertilizers in agriculture in Brazil (IN 61/2020).

BET (Brunauer-Emmet-Teller) surface analysis was performed to compare the surface area (SA), pore volume (PV), and pore size (PS) of the different physical forms. The sample degassing process was carried out at a temperature of 30 °C for 3 hours under vacuum. The analyzes were obtained at a temperature of -196.15 °C. The N adsorption/desorption isotherms were obtained with the Quantachrome© Nova 4000e equipment and the help of NovaWin software, version 11.03. To calculate the SA, the BET multi-point method was used. PV and PS values were calculated by analyzing the desorption curve of the Barrett-Joyner-Halenda (BJH) model.

3.2.3 Site description and soil characterization

The field study was conducted at the Teaching and Research Farm - Cerrado (20° 20´34´´S - 51°23´47´´W), located in the municipality of Selvíria, Mato Grosso do Sul State, with a mean altitude of 357 m. The area belongs to São Paulo State University (UNESP), campus Ilha Solteira city, São Paulo State (Fig. 2). According to Köppen’s classification system, the climate of the region is Aw, with average annual precipitation of 1,200 – 1,500 mm, concentrated from December to March (INMET, 2021).

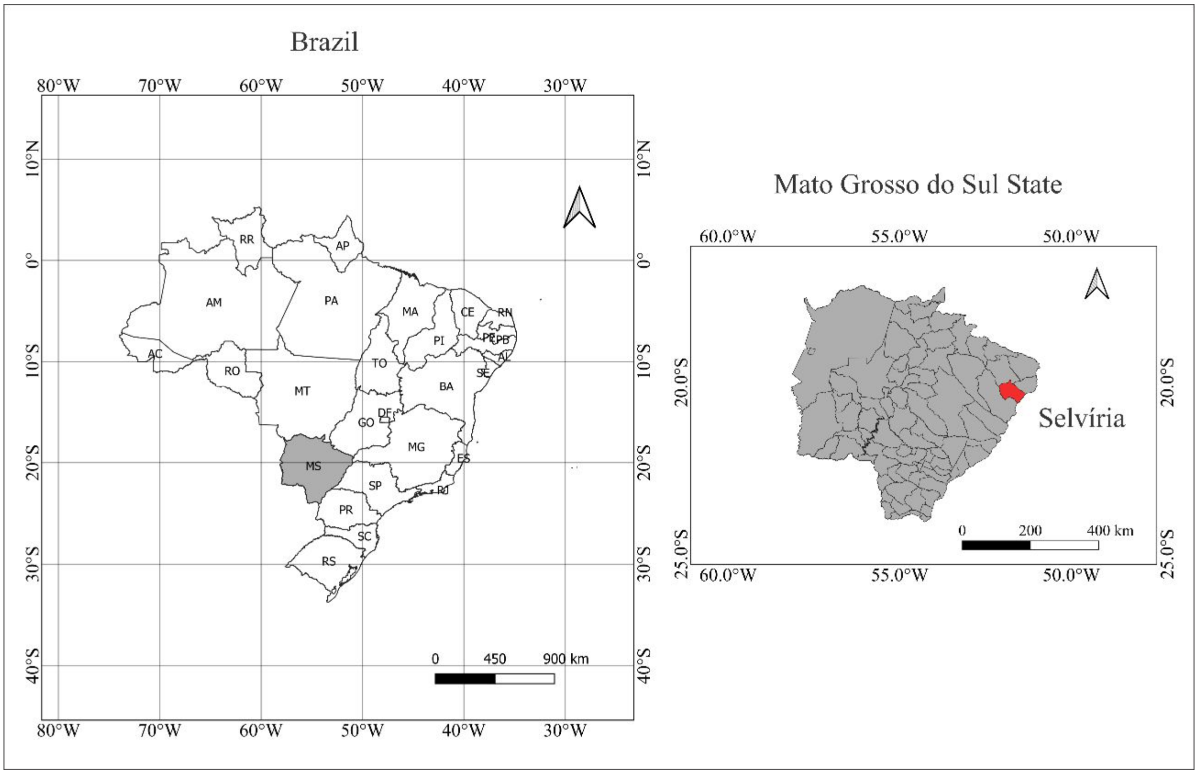

Fig. 2. The geographic location of the experimental area of Selvíria city, Mato Grosso do Sul State, Brazil.

Soil samples were collected from the 0-20 and 20-40 cm layers, air-dried, sieved (2 mm), and stored at environmental conditions. Sub-samples were taken to evaluate soil texture (Teixeira et al., 2017) and fertility parameters: pH (H_2O and $CaCl_2$ 0.01M), exchangeable K^+, $Ca^{2+,}$ and Mg^{2+} and available P (extraction by ion exchange resin and quantification by atomic absorption spectrophotometry), exchangeable Al^{3+} (extraction via KCl and quantification by titration with NaOH), $S\text{-}SO_4^{-2}$ (extraction by phosphate solution calcium and quantification by turbidimetry), Cu^{2+}, Zn^{2+}, Mn^{2+}, Fe^{2+} (DTPA method at pH 7.3 and quantification by atomic absorption spectrophotometry), B (microwave extraction with barium chloride solution and quantification by colorimetry), and organic matter (OM) (combustion) (Raij et al., 2001). The sum of bases (SB), potential cation exchange capacity (CEC, pH 7), base saturation (V%), and aluminum saturation (m%) were calculated (Table 1). The soil was classified as Typic Hapludox (Staff, 2014), as Haplic Ferralsol (Baxter, 2007; Wrb, 2006) with clay texture (~37%).

Table 1. Chemical and physical attributes of the soil of the experimental site.

Attribute	Layer (cm)	
	0-20	20-40
pH (0.01 M $CaCl_2$)	5.2 ± 0.2	5.4 ± 0.1
pH (H_2O)	5.8 ± 0.1	6.0 ± 0.2
OM* (g dm^{-3})	19 ± 1.4	15 ± 0.6
Density (g dm^{-3})	0.97 ± 0.01	0.92 ± 0.01
P (mg dm^{-3})	16 ± 3.7	14 ± 0.6
K ($mmol_c$ dm^{-3})	2.1 ± 0.6	1.8 ± 0.3
Ca^{2+} ($mmol_c$ dm^{-3})	14 ± 1.8	13 ± 0.6
Mg^{2+} ($mmol_c$ dm^{-3})	13 ± 1.0	11 ± 0.0
S-SO_4 (mg dm^{-3})	5 ± 1.0	4 ± 0.6
B (mg dm^{-3})	0.33 ± 0.1	0.12 ± 0.02
Cu (mg dm^{-3})	1.6 ± 0.1	0.9 ± 0.15
Fe (mg dm^{-3})	46 ± 9.0	27 ± 0.6
Mn (mg dm^{-3})	20.6 ± 3.2	10.5 ± 2.0
Zn (mg dm^{-3})	0.9 ± 0.1	0.4 ± 0.06
Al (mmolc dm^{-3})	1 ± 1.0	0 ± 0.0
H+Al* (mmolc dm^{-3})	30 ± 3.8	20 ± 2.0
SB* (mmolc dm^{-3})	29.1 ± 2.8	25.8 ± 0.45
CEC* (mmolc dm^{-3})	59.1 ± 3.9	45.8 ± 1.79
V* (%)	49 ± 5.0	56 ± 3.0
m* (%)	1.69 ± 0.70	0 ± 0
Sandy (g kg^{-1})	550 ± 13	
Silt (g kg^{-1})	80 ± 3	
Clay (g kg^{-1})	370 ± 19	

* OM = organic matter; H+Al = potential acidity; SB = sum of basis; V = base saturation; CEC = cation exchange capacity (pH = 7); m = aluminum saturation.

3.2.4 Experimental design, treatments, and management practices

The experimental design used was randomized blocks, with treatments arranged in a factorial scheme [(3 x 2) + 2], comprising: three physical forms (Pw, powder; G, granule, and Pt, pellets), two P_2O_5-rates of application (70% and 100%, based on soybean needs), plus two additional treatments: *i*) mineral fertilizer (MF) (= 100% of SS-OMF P_2O_5-rate application) and *ii*) absolute control (Ct, without nutrient addition). Four replications were used, totaling 8 treatments and 32 experimental units. The rate of 70% was established based on greenhouse preliminary tests, which allowed the development of soybean plants similar to 100% of MF (Rodrigues et al., 2021). The P_2O_5-needs of the soybean crop was 50 kg ha^{-1} (Table 2) (Raij et al., 1997). To compare the residual P effect of the treatments, they were not reapplied in the maize off-season.

Table 2. Fertilizer application rates (kg ha^{-1} and g $plot^{-1}$) and nutrient equivalent amounts (kg ha^{1}).

Treatment	**P_2O_5 - rate**		**N**	**P_2O_5**	**K_2O**
	kg ha^{-1}	g plot^{-1}	------------------ kg ha^{-1} ----------------		
Control (CT)	0.0	0.0	0.0	0.0	0.0
Mineral fertilizer (MF)					
MAP^a	96.1	115.0	9.6	50.0	0.0
KCl	100.0	108.0	0.0	0.0	60.0
Organomineral fertilizer from sewage sludge (SS-OMF)					
Pw-G-Pt 100%	625.0	674.9	25.0	50.0	50.0
Pw-G-Pt 70%	437.0	472.5	17.5	35.0	35.0

^{a}MAP = monoammonium phosphate. Plot = 12 m²

The plot size was 4 m x 3 m (12 m²), with six lines, spaced 0.45 m. The useful area of each plot consisted of the two central lines, disregarding the 0.5 m border at each end, totaling 2.7 m². All inferences were made considering the useful area, except for soybean nodulation. The width of the smaller skid was 1.7 m and of the biggest skid was 3.5 m. The pre-planting operations (limestone and gypsum application, and soil preparation) were carried out considering soybean planting, followed by maize off-season. The application of dolomitic limestone (PRNT = 90%) was carried out two months (2 t ha^{-1}) and gypsum (850 kg ha^{-1}) fifteen days before soybean planting.

The experiment was implemented on December 7, 2021, in an area with pivot irrigation. The fertilizers (SS-OMFs and MF) were weighed per planting row, manually applied to a depth of 5 cm, and covered with soil. The soybean (TMG 7063 IPRO) planting was carried out mechanically and sowing was adjusted to a depth of about 3 cm. Seed treatment included the use of Masterfix liquid inoculant (2 ml per kg of seed) + Geo CoMo (200 ml ha^{-1}) + Derosal Plus fungicide (carbendazim + thiram, 10 ml of a.i.). After the soybean harvest, the maize off-season (Agroceres AG 7098 PRO2) planting was carried out on April 20, 2022. Seed treatment was performed with Standak® Top (pyraclostrobin + methyl thiophanate) and insecticide (fipronil), considering 10 ml for each 5 kg of seed. The sowing depth was approximately 3 cm over the remaining soybean straw (no-tillage system), adjusted to 3 seeds per meter. At planting, fertilization was carried out with KCl corresponding to 50 kg ha^{-1} of K_2O, and at 30 days after germination, the equivalent of 20 kg ha^{-1} of N in the form of urea was applied in coverage in all treatments, except Ct (Raij et al., 1997). To avoid losses of N by volatilization and promote adequate development, the maize off-season was irrigated with an average depth of 12 mm. For both crops, the harvest was done manually, and the phytosanitary management was carried out according to the technical recommendations for the region (Appendix B).

3.2.5 Sampling and chemical analysis of plant tissue

To evaluate the nutritional status of the plants, the diagnosis leaf was collected in phenological stage R1 (Fig. 2): *i*) for soybeans, 30 newly mature trifoliate were collected per plot, corresponding to the third trifoliate leaf (Fig. 2A) and *ii*) for maize off-season, 10 opposite leaves were collected below the first ear, in the middle third of the leaf, with the midrib, at the stage in which the female inflorescence appears (Fig. 2B) (Raij et al., 1997). The plant samples were placed in paper bags, dried in an oven with forced air circulation at 65°C until constant weight, and ground in a stainless-steel mill with a 1 mm mesh sieve.

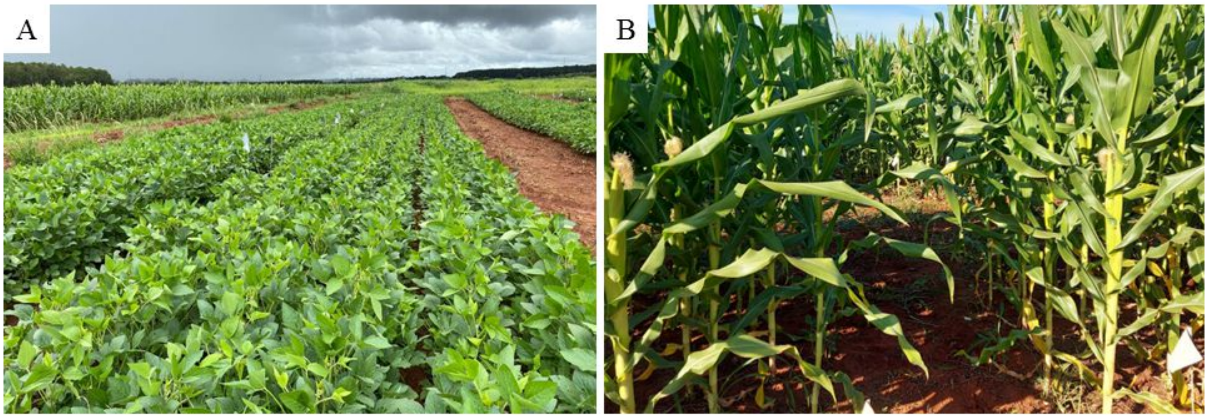

Fig. 2. A) Soybean area and B) maize off-season area, both in the R1 phenological stage (flowering).

Chemical analyses of plant tissue (diagnosis sheet and grains) included the determination of macro (N, P, K, Ca, Mg, and S) and micronutrient (B, Cu, Fe, Mn, and Zn) levels (Malavolta et al., 1997). The semi-total levels of trace elements (whole plant and grains) (As, Cd, Cr, Cu, Hg, Fe, Mn, Ni, Se, and Zn) were determined by adapting the EPA 3051a method (USEPA, 2007). For this, 0.5g of plant samples were weighed and digested in nitric acid, with pre-digestion performed using 2 ml of ultrapure hydrogen peroxide (H_2O_2) solution and volume up to 25 ml. Readings were performed by inductively coupled plasma mass spectrometry (ICP-OES). The nutrient accumulation (extraction) in both crops was estimated by:

Nutrient accumulation (g or kg ha^{-1}) = [grain mass (kg ha^{-1})] x [nutrient in grain mass (g or mg kg^{-1})] + [shoot dry mass (kg ha^{-1})] x [nutrient in shoot dry mass g or mg kg^{-1})]

3.2.6 Vegetative development and productivity

At the end of each crop cycle, 10 plants of the useful area were randomly chosen for biometric evaluations: *i*) Soybean: shoot dry mass (SDM, kg ha^{-1}), where plants were cut close to the ground, dried in an oven with forced air circulation at 65°C until constant weight, crushed and weighed, disregarding grains; Nodulation, considering the presence of active nodulation (AN) by removing roots from the border lines of the plots, washed in a current borderline and the nodules were cut to verify the presence of leghemoglobin; *ii*) Maize off-season: SDM (kg ha^{-1}), where plants were cut close to the ground, dried in an oven with forced air circulation at 65°C until constant weight, crushed and weighed, considering the dry mass of the cob, but disregarding grains dry mass. To determine productivity, the total mass of grains of both crops was mechanically threshed, weighed, and the moisture was adjusted to 13%. The results were extrapolated to kg ha^{-1}.

3.2.7 Agronomic efficiency index

To evaluate the crop productivity of organomineral treatments compared to MF (MAP plus KCl), the agronomic efficiency index (AEI) was calculated considering the equivalent rate of application (with 100% P_2O_5-needs) (Frazão et al., 2018; Grohskopf et al., 2019), using the following equation:

$$\text{AEI (\%)} = [(P_{SS\text{-}OMF} - P_{Ct}) / (P_{MF} - P_{Ct})] \times 100$$

$P_{SS\text{-}OMF}$ = Productivity (kg ha^{-1}) in SS-OMF treatments
P_{Ct} = Productivity (kg ha^{-1}) in Ct treatment, without P addition.
P_{MF} = Productivity (kg ha^{-1}) in MF treatment.

3.2.8 Sampling and chemical analysis of soil

At the end of both crop cycles, 0-20 cm composite soil samples were collected, considering 4 points (2 in the planting line and 2 between the lines) within the useful area of each plot. In these samples, the same chemical fertility attributes, and semi-total levels of trace elements (As, Cd, Cr, Cu, Fe, Mo, Mn, Ni, Se, and Zn) initially determined in the soil (item 3.2.3), were quantified, following the same procedures.

3.2.9 Sampling and biological analysis of soil

After each crop harvest, to verify the effects of using SS-OMF on soil quality, composite 0-10 cm soil samples were collected, consisting of 4 random points in the planting row of each plot. Samples were stored at 4°C in a cold chamber. To avoid contamination, soil sampling was carried out with a Dutch auger which was cleaned with a 0.01% v/v sodium hypochlorite solution between plots.

Microbial biomass carbon quantification (MBC) was performed using the indirect Fumigation-Extraction method (Vance et al., 1987). The determination of the enzyme's β-glucosidase (βG), acid phosphatase (Ap), and arylsulfatase (As) activities, respectively associated with the C, P, and S soil cycles, were carried out (Tabatabai, 1994). The determination of easily extractable glomalin (EEG) was performed (Wright and Upadhyaya, 1998) with quantification via spectrophotometry (Biochrom EZ Read 400) with a wavelength adjusted to 595 nm. Each sample was subjected to three readings and the EEG values were expressed based on a standard curve (Bradford, 1976).

3.3 Data analysis

The data were submitted to analysis of variance (Appendix D) and, when the F test was significant, the unfolding of the interaction between the factors (physical forms and rates) was carried out. Verification of data normality was performed using the Shapiro-Wilk and Bartlett tests. When the data normality criteria were not met, adequate transformation proceeded. To compare the means of physical-chemical attributes of the organomineral fertilizer's physical forms, structural characteristics, and AEI (agronomic efficiency index), the Tukey test $p<0.05$) was applied. Means were compared by Tukey's test at a significance level of 5% and additional treatments were compared using Dunnett's test ($p<0.05$). All analyzes were performed using the R Studio software (RStudio, 2020).

3.4 Results and discussion

3.4.1 SS and SS-OMF´s environmental safety and characterization

The SS used in this study meets the legal requirements regarding the presence of pathogenic agents (E. Coli = 3×10^2 CFU g^{-1}), being classified as class A (Brazil, 2020a). The drying and grinding of raw SS were capable to reduce the pathogenic load of this waste (Oliveira et al., 2018; Rodrigues et al., 2021; Santos et al., 2020). The number of viable helminth eggs in the SS-OMF was 0.05 eggs g^{-1} of total solids, 20 times less than the limit considered safe for handling in Brazil (Brazil, 2020b). Furthermore, Thermotolerant Coliforms and *Salmonella* sp were not detected in the SS-OMF. Similar results were obtained with the synthesis of an SS-OMF mixed with poultry litter ash that was free of the presence of pathogens, once the drying process, as well as the increase in the osmotic pressure caused by the addition of mineral sources, are efficient in eliminating the presence of potential pathogenic agents (Kominko et al., 2021).

The concentrations of heavy metals found in SS do not bring any restriction of use concerning the safety limits established by the Brazilian legislation, being classified as Class 1 (Brazil, 2020a) (Table 3). Similarly, the levels of heavy metals in the SS-OMF were below the maximum limit (Normative instruction N° 7, annex 5) (Brazil, 2016) except for Ni (Table 3). The maximum limits allowed for Ni for organomineral fertilizers are being reviewed in Brazil, since this element is currently considered a micronutrient for plants, especially for soybeans, due to its role in plant nodulation and activity of the urease enzyme (Lavres et al., 2016; Levy et al., 2019; Yusuf et al., 2011). Added to this, the SS-OMFs formulations reduce the accumulation of heavy metals in the soil compared to the strict use of SS, since there is a dilution of these elements in the SS-OMF manufacture (Kominko et al., 2017a). Also, the higher nutrient concentration of SS-OMFs promotes lower field application rate needs (Antille et al., 2013; Pawlett et al., 2015). For example, in this study, the SS application rate would be 2200 kg ha^{-1}, compared to only 625 kg ha^{-1} of SS-OMF, a 3.5-fold reduction.

Table 3. Heavy metals content (mg kg^{-1} in dry weight basis) on sewage sludge (SS) and organomineral fertilizer from sewage sludge (SS-OMF) in three physical forms and threshold values settled by Brazilian (CONAMA 498/2020 and Normative Instruction 7/2016) legislations for agricultural use.

Element	**SS**	**SS-OMF**			**Classification** [a]	
		Powder (Pw)	**Granule (G)**	**Pellet (Pt)**	**Conama 498/2020**	**NI 7/2016**
As	1.74	4.6 ± 0.37	3.9 ± 0.45	4.1 ± 0.48	41	751
B	44.7	-	-	-	NR	NR
Ba	454	-	-	-	1300	NR
Cd	0.99	4.4 ± 0.09	3.2 ± 0.05	3.3 ± 0.18	39	56.5
Cr	379	-	-	-	1000	NR
Cr^{6+}	0.63	<0.7	<0.7	<0.7	NR	2.0
Cu	431	-	-	-	1500	NR
Fe	23000	-	-	-	NR	NR
Hg	0.85	0.48 ± 0.08	0.41 ± 0.01	0.44 ± 0.03	17	15.7
Mn	144	-	-	-	NR	NR
Mo	4.8	-	-	-	50	NR
Ni	108	70.1 ± 2.5	86.0 ± 4.1	86.9 ± 3.3	420	70.0
Pb	30.4	17.5 ± 0.66	20.6 ± 0.35	19.1 ± 0.61	300	1382.5
Se	< 1	<0.5	<0.5	<0.5	36	80.0
Zn	1690	-	-	-	2800	NR

[a] CONAMA – Environment National Council (limits for sewage sludge) and MAPA – Ministry of Agriculture, Livestock and Supply (limits for organomineral fertilizers [As, Cd, Pb and Hg limits were calculated according to the limits for each element {appendix V} plus the %P_2O_5 guarantees {appendix I, column A} and guaranteed micronutrients {appendix I, column B}] and limits for Ni, Zn and Cr^{6+} are unchanged [appendix V]).

The characterization of physicochemical parameters confirmed the similarity of the SS-OMF physical forms evaluated and the proposed formulation 4-8-8 (Pw, powder; G, granule; and Pt, pellet) met the requirements established by Brazilian legislation (IN 61/2020) (Fig. 3 and Table 4). The average diameter of the granules ranged from 1-3 mm. Pelletizing SS-OMF generated adequate pellets, with uniform size, good consistency, and hardness resulting in pellets with an average length of 11 mm and diameter of 3 mm (Fig. 3C). Pampuro et al. (2017) reported that the pressure during the pelleting of organic materials, such as pork paste added with wood sawdust, does not influence the physicochemical properties of the pellets, and the particle size is a critical aspect to obtaining pellets with desirable mechanical properties such as high density. The operational easiness and reproducibility make the OMF pelletizing process attractive (Grafmüller et al., 2022).

Fig. 3. Organomineral fertilizer from sewage sludge in the physical forms of powder (A), granule (B), and pellet (C).

Table 4. Physical-chemical attributes of the sewage sludge (SS) and organomineral fertilizer from sewage sludge (SS-OMF) in three distinct physical forms.

Attributes	**SS**	**SS-OMF**			
		Powder (Pw)	**Granule (G)**	**Pellet (Pt)**	**Mean**
pH ($CaCl_2$ 0.01 M)	4.9	4.7 ± 0.11 a	4.7 ± 0.10 a	4.6 ± 0.10 a	5.3
pH (H_2O)	5.4	5.3 ± 0.09 a	5.4 ± 0.11 a	5.3 ± 0.10 a	4.7
Density ^{a} (g cm$^{-3)}$	0.93	0.89 ± 0.01 a	0.89 ± 0.01 a	0.87 ± 0.01 a	0.89
Moisture ^{a} (%)	6.1	2.0 ± 0.14 a	2.1 ± 0.00 a	1.9 ± 0.35 a	2.0
Organic matter (%)	62.6	43.2 ± 0.61 a	43.9 ± 1.1 a	43.0 ± 0.21 a	43.4
Organic carbon (%)	33.6	24.0 ± 0.31 a	24.4 ± 0.61 a	23.9 ± 0.11 a	24.1
N total (%)	5.50	4.1 ± 0.15 a	4.1 ± 0.07 a	4.0 ± 0.10 a	4.1
C/N ^{a}	6	6 ± 0.00 a	6 ± 0.00 a	6 ± 0.00 a	6
P_2O_5 total (%)	3.0	10.2 ± 0.68 a	8.8 ± 0.19 a	10.0 ± 0.19 a	9.6
P_2O_5 Soluble ^{b} (%)	2.6	9.7 ± 0.98 a	8.1 ± 0.78 a	9.0 ± 0.29 a	8.7
K_2O (%)	0.13	9.8 ± 0.21 a	8.0 ± 0.12 a	8.0 ± 0.36 a	8.6
K_2O Soluble ^{b} (%)	-	9.2 ± 0.46 a	8.9 ± 0.69 a	8.6 ± 0.73 a	8.9
Ca (%)	1.94	2.9 ± 0.05 a	2.9 ± 0.23 a	3.0 ± 0.18 a	2.9
Mg (%)	0.34	1.9 ± 0.03 a	1.8 ± 0.05 a	1.8 ± 0.09 a	1.9
S (%)	0.10	0.57 ± 0.07 a	0.58 ± 0.07 a	0.61 ± 0.0 a	0.59
B (mg kg^{-1})	-	273.3 ± 25.1 a	246.7 ± 5.8 a	240.0 ± 20.0 a	253.3
Cu (mg kg^{-1})	-	163.6 ± 5.8 a	180.0 ± 10.0 a	173.3 ± 15.3 a	172.2
Fe (g kg^{-1})	-	12.64 ± 0.47 a	14.12 ± 1.24 a	14.14 ± 0.75 a	13.64
Mn (mg kg^{-1})	-	346.7 ± 11.5 a	333.3 ± 15.3 a	340.0 ± 17.3 a	340.0
Zn (mg kg^{-1})	-	626.7 ± 35.1 a	710.0 ± 70.0 a	680.0 ± 36.6 a	672.2
CEC ^{c} (mmol$_c$ kg^{-1})	380	180.0 ± 20.0 a	183.3 ± 5.8 a	153.3 ± 32.1 a	172.2
WHC ^{c} (%)	78.7	95.7 ± 1.2 b	94.7 ± 1.2 b	112.0 ± 1.6 a	100.8

EC [c] (mS cm^{-1})	7.35	69.9 ± 5.4 a	58.7 ± 4.2 a	62.3 ± 5.9 a	63.9

[a] Wet base values. [b] extracted using neutral ammonium citrate plus water. [c] CEC = cation exchange capacity; WHC = water holding capacity; and EC = electric conductivity. Means followed by the same letter in columns do not differs according to the Tukey´s test ($P<0.05$)

The OM content decreased by about 30% and OC by about 28%, which led to a lower CEC in the SS-OMF compared to the use of SS (Table 4). Considering the OM content of SS-OMF (~43%) and its application rate in the soil (625 kg ha^{-1}), it is estimated that there is an increase of approximately 0.08% in the soil MO content. This shows that the benefits associated with increasing soil OM with the use of organominerals require continuous long-term applications, especially in tropical regions where soil microbial activity is capable of converting 2/3 of the applied OM into CO_2 (Normand et al., 2021). Compared to the raw SS, the SS-OMF had higher P_2O_5 total, P_2O_5 soluble, and K_2O total concentrations, in the order of 3, 3, and 66 times, respectively (Table 4). N total content decreased by approximately 25% (Table 4). In a preliminary study in a greenhouse using the same SS-OMF in soybean, no negative impacts on nodulation were seen, with plants showing proper vegetative development as mineral treatment (Rodrigues et al., 2021). Considering the SS-OMF application rate, meaning an input of about 25 kg of total N, corresponding to approximately 8 kg ha^{-1} of N-mineral. This amount is less than what is considered harmful for adequate soybean nodulation (20-30 kg ha^{-1}), and consequent N biological fixation, since about 50-60% of the N required by the crop comes from biological fixation (Cordeiro and Echer, 2019).

Regarding the morphological characteristics of the different physical forms of SS-OMF, the Pw showed a higher superficial area (AS) pore size (PS) and pore volume (PV), and a predominance of mesopores (2-50 nm) (Table 5 and appendix C). This indicates that Pw can have a greater ability to supply nutrients to plant roots in the short term and to release carbon to soil microorganisms from the organic fraction compared to G and Pt. On the other hand, Pt presents a greater adsorption potential (nutrients, potentially toxic elements, and water), thus showing a greater residual effect in soil (Table 5 and Appendix C). This can be a desirable effect in the case of succession crops such as maize off-season, grown right after the soybean harvest, which is very common in Brazil (Bolfe et al., 2020; Carneiro and Costa, 2016). Moreover, as expected, Pt showed a higher water-holding capacity (Table 4), indicating storing water molecules for a longer time, with the potential to reduce water stress in rainfed crops (Batista et al., 2018). The ability to adsorb nutrients is greater in materials with a predominance of micropores (<2 nm); the ability to allow microbial microcosms for their survival promoted by mesopores (2-50 nm); and, high aeration, water infiltration, and habitat for microorganisms is associated with the presence of macropores (>50 nm) (Fachini et al., 2021; Wong and Ogbonnaya, 2021). The PV reflects the permeability of water (mass flow of water) and its flow velocity through the material, therefore, the greater the number of pores, the greater the porosity and the lower the density (Hermawan et al., 2019).

Table 5. Structural characteristics of an organomineral from sewage sludge (SS-OMF) in three physical forms (Pw, powder; G, granule; Pt, pellet).

SS-OMF physical form	Surface area	Pore volume	Pore size
	m^{-2} g	cm^{-3} g	Å
Pw	1.95 ± 0.038 a	0.0057 ± 0.0002 a	20.7 ± 0.80 a
G	1.53 ± 0.072 b	0.0041 ± 0.0000 b	17.4 ± 2.08 ab
Pt	1.05 ± 0.025 c	0.0028 ± 0.0006 c	15.3 ± 0.02 b

Means followed by the same letter in columns do not differ according to Tukey´s test ($P<0.05$).

3.4.2 Soybean crop

3.4.2.1 Nutritional status and nutrient accumulation

The SS-OMF in different physical forms and P_2O_5-rates of application had similar effects on nutritional status in soybean compared to MF treatment (Table 6). The nutritional status of macro and micronutrients in the diagnosis leaves was compatible with the ranges considered adequate (Barbosa et al., 2016a; Raiesi and Khadem, 2019; Raij et al., 2022). Possibly, the contrasts between treatments were attenuated due to the low responsiveness of the soil in the experimental area (Table 1), considering that only one application of SS-OMF was carried out (Barbosa et al., 2014; Raiesi and Khadem, 2019). Only B and Cu foliar contents were significantly ($p<0.05$) influenced by treatments, but with no differences between SS-OMFs and MF (Table 6). The B level in MF (48.2 mg kg^{-1}) was higher compared to Ct (35.5 mg kg^{-1}) (Table 4), probably due to the restriction of other nutrients in Ct, which limited root development and B-absorption by plants (Vera et al., 2021). The Cu level showed only a rate effect, with a higher average level at the 100%-P_2O_5 (22.0 mg kg^{-1}) when compared to 70%-P_2O_5 (15.6 mg kg^{-1}) due to the higher input at the 100% SS-OMF rate (172 mg kg^{-1}) than at the 70% rate (120 mg kg^{-1}) (Table 4) (Kumar et al., 2019).

Table 6. Macro (N, P, K, Ca, Mg, and S) (g kg^{-1}) and micronutrients content (B, Cu, Fe, Mn, and Zn) (mg kg^{-1}) in the soybean diagnosis leaf amended with an organomineral fertilizer in three physical forms [powder (Pw), pellet (Pt), and granule (G)], two P_2O_5 rates of application (70% and 100%) and two additional treatments (MF = mineral fertilizer and Ct = without fertilization).

Treatment	**N**		**P**		**K**		**Ca**		**Mg**		**S**	
	---------------------- P_2O_5-rate (%) ----------------------											
Physical form	70	100	70	100	70	100	70	100	70	100	70	100
Pw	48.3		3.5		22.0		8.3		5.8		2.8	
G												
Pt												
MF	49.4		3.5		22.8		8.5		5.9		2.7	
Ct	48.3		3.3		20.9		8.2		5.8		2.6	
Sufficiency range*	40-54		2.5-5		17-25		4-20		3-10		2.1-4	

Treatment	**B**		**Cu**		**Fe**		**Mn**		**Zn**	
	---------------------- P_2O_5-rate (%) ----------------------									
Physical form	70	100	70	100	70	100	70	100	70	100
Pw	43.7		15.6 B	21.9 A	125.1		68.8		45.1	
G										
Pt										
MF	48.2		15.7		136		70.0		42.1	
Ct	35.5		19.3		131		67.7		38.5	
Sufficiency range*	21-55		10-30		50-350		20-100		20-50	

* (Raij et al., 2022). Distinct lowercase letters indicate a difference between SS-OMF physical forms and distinct capital letters indicate a difference between SS-OMF application rates by Tukey's test ($p < 0.05$). SS-OMF mean accompanied by ° differs from additional treatment MF and the mean accompanied by * differs from additional treatment Ct (Dunnett, $p<0.05$). Ct means accompanied by ▲ differs from additional treatment MF (Dunnett, $p<0.05$). Mean values (n=4).

Except for Cu, the accumulation of macro and micronutrients in soybean plants was similar between SS-OMFs and MF treatments (Table 7). The use of Pt100 (194 g ha^{-1}) promoted greater Cu accumulation in soybean plants than MF (118 g ha^{-1}) (Fig. 5). In soil, Cu adsorption is preferentially regulated by the presence of MnO > soil organic matter > Fe_2O_3 > Al^{3+} > clay minerals (Abat et al., 2012). However, in Oxisols with a low amount of Cu in solution, the complexation with organic acids present in the SS makes this element more easily absorbable (Dalpisol et al., 2017). Due to its immobilization in the soil, root interception is crucial for plant uptake, and good root development promotes better access to Cu (De Conti et al., 2016; Jin et al., 2017). This can occur in Pt, given that AN was greater in this treatment (Moreira et al., 2022).

Regarding the SS-OMFs treatments, there was a significant difference depending on the physical forms in the accumulation of P, Ca, S, and B, in which the physical form Pt promoted the greatest accumulation of these elements compared to G and Pw, however, without significantly differing of the MF and Ct treatments (Table 7). The slower release of nutrients in Pt compared to G and Pw may have favored nutrient uptake by soybean (Fachini et al., 2021). The organic fraction protects the solubilized P from the mineral fraction of OMFs against fixation with iron and aluminum oxides present in soils, being an interesting alternative to succession crops (Martins et al., 2017) mainly in clayey tropical soils with high kaolinite and metal hydrous oxide content, which favor P sorption and fixation, reducing its availability and uptake by plants (Campos et al., 2018; Mackay et al., 2017; Rodrigues et al., 2021). The accumulation of N, Mg, Mn, and Zn was influenced only as a function of the SS-OMF application rate, where the 100% promoted greater accumulation than the 70% (Table 7) due to the higher amount of nutrients added.

Table 7. Accumulated levels of macro-N, P, K, Ca, Mg, S (kg ha^{-1}) and micronutrients B, Cu, Fe, Mn, and Zn (g ha^{-1}) in soybean aerial part amended with an organomineral fertilizer in three physical forms [powder (Pw), pellet (Pt), and granule (G)], two P_2O_5 rates of application (70% and 100%) and two additional treatments (MF = mineral fertilizer and Ct = without fertilization).

Treatment	**N**		**P**		**K**		**Ca**		**Mg**		**S**	
	---------------------- P_2O_5-rate (%) ----------------------											
Physical form	70	100	70	100	70	100	70	100	70	100	70	100
Pw	222 B	268 A	152 b		161		46 b		32.5B	37.7A	22.0 b	
G			161 b				53 ab				24.2 ab	
Pt			207 a*				56 a				26.1 a*	
MF	237		183		161		52		34.4		23.5	
Ct	224		145▲		135		45		31.8		19.6	

Treatment	**B**		**Cu**		**Fe**		**Mn**		**Zn**	
	---------------------- P_2O_5-rate (%) ----------------------									
Physical form	70	100	70	100	70	100	70	100	70	100
Pw	255 b		86 aB	152 bA	954		383 B	476 A	314 B	371 A
G	312 ab		108 aA	136 bA						
Pt	328 a		91 aB	194 aA°*						
MF	312		118		1122		420		345	
Ct	249		122		899▲		442		293	

Distinct lowercase letters indicate a difference between SS-OMF physical forms and distinct capital letters indicate a difference between SS-OMF application rates by Tukey's test ($p < 0.05$). SS-OMF mean accompanied by ° differs from additional treatment MF and the mean accompanied by * differs from additional treatment Ct (Dunnett, $p<0.05$). Ct means accompanied by ▲ differs from additional treatment MF (Dunnett, $p<0.05$). Mean values (n=4).

2.4.2.2 Heavy metal content in the soil-plant system

The range of heavy metals contents in the soil and plant grains after harvesting soybean were much lower than those considered harmful to soil quality (Cetesb, 2021) and for human and animal consumption, according to Brazilian normative (ABIA, 1991) and international regulation (FAO/WHO, 2001) (Appendix E). In the grains, the contents of Cd, Ni, Pb, Mo, Se, and Hg were lower than the limit of quantification, and in the soil, the same was observed for the elements As, Cu, Hg, Ni, Mo, and Se. One of the major concerns about the use of SS-based fertilizers is the possibility of soil-water-plant contamination by heavy metals (Kominko et al., 2017a; Tytła, 2019). Here, the proposed SS-OMF formulation increased the concentration of nutrients from mineral sources, which allowed the dilution of heavy metals once the application rate is lower than the use of SS in the soil. Our findings are in line with those obtained in other studies evaluating the possible soil and plant contamination by heavy metals from SS-OMF (Antille et al., 2017; Deeks et al., 2013; Kominko et al., 2019; Rodrigues et al., 2021).

There was no effect of physical forms or P_2O_5-rate of SS-OMF on heavy metal content in the soil, except for Zn, where the 70% rate (7.45 mg kg^{-1}) showed a higher semi-total Zn content compared to the 100% rate (6.65 mg kg^{-1}) and the MF treatment (5.85 mg kg^{-1}) (Table 8). In soybean grains, there was a difference in the levels of Cu, only about the additional treatments, where MF (13.0 mg kg^{-1}) was significantly higher than Ct (12.1 mg kg^{-1}) (Table 8). For Fe, the physical form Pt (104 mg kg^{-1}) promoted significantly lower Fe contents in grains compared to Pw (143 mg kg^{-1}) and Ct (144 mg kg^{-1}) (Table 8). Our results indicate that a single application of SS-OMF at rates considered adequate was not enough to increase the levels of heavy metals in soil and soybeans, regardless of the physical form used. Similar findings were observed with the application of an SS-OMF plus poultry litter in a pot trial with sunflower and maize, where increasing rates of application promoted greater accumulation of metals, mainly Cd and Ni, but at volume levels considered low (Kominko et al., 2022).

Table 8. Levels of heavy metals in the soil and soybean grains ($mg\ kg^{-1}$) amended with an organomineral fertilizer in three physical forms [powder (Pw), pellet (Pt), and granule (G)], two P_2O_5 rates of application (70% and 100%) and two additional treatments (MF = mineral fertilizer and Ct = without fertilization).

SOIL

Treatment	Cd		Cr		Fe		Mn		Pb		Zn	
	---------------------- P_2O_5-rate (%) ----------------------											
Physical form	70	100	70	100	70	100	70	100	70	100	70	100
Pw	0.58		35.5		28525		5.95		0.47		7.45 A°	6.65 B
G												
Pt												
MF	0.50		36.3		26362		5.85		0.49		5.85	
Ct	0.62		32.4		30836		6.35		0.50		7.07	

GRAINS

Treatment	Cu		Fe		Mn		Zn	
	---------------------- P_2O_5-rate (%) ----------------------							
Physical form	70	100	70	100	70	100	70	100
Pw	12.2		143 a		25.8		43.0	
G			105 b					
Pt			104 b*					
MF	13.0		142		26.8		44.0	
Ct	12.1▲		144		25.7		42.8	

Distinct lowercase letters indicate a difference between SS-OMF physical forms and distinct capital letters indicate a difference between SS-OMF application rates by Tukey's test ($p < 0.05$). SS-OMF mean accompanied by ° differs from additional treatment MF and the mean accompanied by * differs from additional treatment Ct (Dunnett, $p<0.05$). Ct means accompanied by ▲ differs from additional treatment MF (Dunnett, $p<0.05$). Mean values (n=4).

3.4.2.3 Bioindicators of soil quality

The acid phosphatase (Ap) enzyme activity was significantly ($p<0.05$) influenced by the physical forms of the SS-OMF (Table 9). Lower Ap activity was found in the SS-OMF Pt physical form (118 mg *p*NF kg^{-1} soil hour^{-1}) compared to Pw (159 mg *p*NF kg^{-1} soil hour^{-1}), G (160 mg *p*NF kg^{-1} soil hour^{-1}) and MF (180 mg *p*NF kg^{-1} soil hour^{-1}) treatments showing that, at least in the short term (soybean crop), the access of P by the soil microbiota is more important than the amount of P added (Table 9). The Ap is an extracellular enzyme synthesized both by plants and soil microorganisms, mainly fungi (Rejsek et al., 2012). Our results indicate that the smaller contact surface area and pore volume in Pt physical form (Table 5) may have hindered access to organic-P in the pellet (Pampuro et al., 2017; Romano et al., 2014). In response, the Ap activity in the soil was higher in the MF, Pw, G, and Ct treatments, in which there is relative P availability (Ch'ng et al., 2017) since it is important to consider that the soil of this experiment contains levels of P considered medium (16 mg dm^{-3}, 0-20 cm; and 14 mg dm^{-3}, 20-40 cm) (Table 1).

A similar tendency to As can be observed in the activity of the enzyme aryl sulfatase (As), mainly at the 70% rate, where the SS-OMF Pt physical form (24.7 mg *p*NS kg^{-1} soil hour^{-1}) and the MF (31.3 mg *p*NS kg^{-1} soil hour^{-1}) showed significantly ($p<0.05$) lower activity of this enzyme than Pw70 (41.4 mg *p*NS kg^{-1} soil hour^{-1}), Pw100 (41.1 mg *p*NS kg^{-1} soil hour^{-1}), G70 (42.2 mg *p*NS kg^{-1} soil hour^{-1}), G100 (40.9 mg *p*NS kg^{-1} soil hour^{-1}) and Ct (41.3 mg *p*NS kg^{-1} soil hour^{-1}) (Table 9). Our findings suggest a trend that greater access to organic S (Pw and G) promoted greater As activity. On the other hand, the high activity of As in Ct reflects the microbiota adapted to conditions of nutritional restriction, confirming that enzymatic synthesis in stressful situations is high (Dotaniya et al., 2019; Utobo and Tewari, 2015). The As is an intra/ extracellular enzyme that catalyzes the hydrolysis of organic sulfate esters to S-SO_4^{-2}, synthesized by soil microorganisms, animals, and plants (Chen et al., 2019; Siwik-Ziomek et al., 2016). Generally, the increase in As activity is associated with the addition of OM to the soil due to the addition of S organic (Sobucki et al., 2021). However, the effects of OMF fertilization in tropical soils are poorly investigated, once there is the presence of organic and inorganic S in the formulation, in which possible synergistic or inhibitory effects on soil biological indicators are still unknown (Andreote et al., 2014).

No significant effect ($p<0.05$) was observed on beta-glucosidase (βG) activity (mean = 61.4 mg *p*NP kg^{-1} soil hour^{-1}), carbon of microbial biomass (CBM) (mean = 511 mg C kg^{-1} soil) and easily exchangeable glomalin (EEG) (mean = 3.22 mg ml^{-1}) after soybean cultivation

(Table 9). The lack of significant effects in βG and CBM agrees with the results obtained in our previous study with the same SS-OMF and MFs, confirming that no suppressive effects were observed in biological indicators of soil quality as a function of the use of SS-OMFs in the short term (Rodrigues et al., 2021).

Table 9. Enzyme activities of beta-glucosidase (βG, mg *p*NF kg^{-1} soil hour^{-1}), acid phosphatase (Ap, mg *p*NF kg^{-1} soil hour^{-1}), arylsulfatase (As, mg *p*NS kg^{-1} soil hour^{-1}), easily exchangeable glomalin (EEG, mg ml^{-1}) and carbon of microbial biomass (CBM, mg C kg^{-1} soil) in the soil after soybean harvest amended with an organomineral fertilizer in three physical forms [powder (Pw), pellet (Pt), and granule (G)], two P_2O_5 rates of application (70% and 100%) and two additional treatments (MF = mineral fertilizer and Ct = without fertilization).

Treatment	**βG**		**Ap**		**As**		**EEG**		**CBM**	
	---------------------- P_2O_5-rate (%) ----------------------									
Physical form	70	100	70	100	70	100	70	100	70	100
Pw	61.4		159 a		41.4 aA°	41.1 aA°	3.22		510	
G			160 a		42.2 aA°	40.9 aA°				
Pt			118 b°		24.7 bB*	35.5 aA				
MF	54.7		180		31.3		3.21		470	
Ct	63.3		150		41.3▲		2.97		540	

Distinct lowercase letters indicate a difference between SS-OMF physical forms and distinct capital letters indicate a difference between SS-OMF application rates by Tukey's test ($p < 0.05$). SS-OMF mean accompanied by ° differs from additional treatment MF and the mean accompanied by * differs from additional treatment Ct (Dunnett, $p<0.05$). Ct means accompanied by ▲ differs from additional treatment MF (Dunnett, $p<0.05$). Mean values (n=4).

3.4.2.4 Soil nutrients and fertility parameters

The use of SS-OMF had little and inconsistent effect on soil nutrient contents and fertility parameters (Table 10 and Appendix D). This attenuated response to the first year of cultivation is because the soil in this study has a high clay content, being little responsive to management (Table 1) (Antonangelo et al., 2019).

The S and Fe levels were higher with the use of the physical form Pw at a 100% rate compared to G, Pt, MF, and Ct (Table 10). Tropical clayey soils with high clay content, as observed in this study, rarely require S fertilization being often neglected, which puts in check the development of crops with high agronomic potential that require greater amounts of S in the long-term (Pias et al., 2019). Studies carried out in Brazil, the United States, and Argentina indicates soils with S deficiency in soybean areas, which can lead to decreases in crop productivity, since S is a determinant in protein synthesis, mainly in the amino acid's methionine and cysteine (Barbosa et al., 2016b; Das and Dkhar, 2012). Possibly the addition of SS-OMF, mainly in the Pw100 treatment, acted to increase the available levels of S, which in turn acted on the availability of Fe in the soil solution Plants improved Fe uptake when there is the presence of S in the soil solution since S modulates the expression of specific transporters in the roots of plants that act in the availability of Fe (Astolfi et al., 2021). Also, the use of SS-OMFs can stimulate microorganisms associated with the solubilization of S, there may have been stimulation of siderophore microorganisms which secrete Fe-chelating molecules and increase Fe availability through its solubilization (Schiessl et al., 2017). It is confirmed by the greater activity of the enzyme As (Table 9) in the function of SS-OMF physical forms which may have been favored by the greater contact surface in Pw.

Concerning the Fe increase in Pw, it is important to highlight that the Fe content is associated with SS since all the Fe comes from the organic matrix. The organic fraction (SS) contains humic substances that can delay the crystallization processes of Fe and act in the formation of water-soluble Fe complexes, improving Fe soil movement and plant uptake favored in Pw physical form, due to the greater contact surface of the particles with the soil (Alvarenga et al., 2015; Usman et al., 2012; Zanin et al., 2019). It can promote greater intensity in biochemical reactions and Fe access by plant roots in the short term (Fachini et al., 2021; Mieldazys et al., 2017; Wang et al., 2021).

The evaluated fertility parameters did not show a significant effect ($p<0.05$) as a function of the treatments (Appendix D) that ranged: OM = 9.3 – 11.3 g dm^{-3}, CEC = 58 – 65 $mmol_c\ dm^{-3}$, pH = 5.60 – 5.80, base saturation percentage (V%) = 79.7 – 82.5, potential acidity (H+Al) = 10.5 – 11.0, and the sum of basis (SB) = 49.9 – 52.4 $mmol_c\ dm^{-3}$.

Table 10. Available levels of macro-K, Ca, Mg, ($mmol_c$ dm^{-3}), P and S (mg dm^{-3}) and micronutrients B, Cu, Fe, Mn and Zn, (mg dm^{-3}) in soil (0-20 cm) after soybean harvest amended with an organomineral fertilizer in three physical forms [powder (Pw), pellet (Pt), and granule (G)], two P_2O_5 rates of application (70% and 100%) and two additional treatments (MF = mineral fertilizer and Ct = without fertilization).

Treatment	P		K		Ca		Mg		S	
	---------------------- P_2O_5-rate (%) ----------------------									
Physical form	70	100	70	100	70	100	70	100	70	100
Pw	22.8		2.5		34.0 a		12.8		6.57 aB	9.30 aA°*
G					28.3 b				5.00 abA	6.67 bA
Pt					35.0 a				4.33 bA	5.68 bA
MF	20.0		2.6		32.2		12.7		4.67	
Ct	19.7		2.2		32.0		13.0		5.50	

Treatment	B		Cu		Fe		Mn		Zn	
	---------------------- P_2O_5-rate (%) ----------------------									
Physical form	70	100	70	100	70	100	70	100	70	100
Pw	0.13 bA	0.13 aA	1.33		12.5 aB	17.3 aA°*	5.95		0.53 aA	0.60 bA
G	0.25 aA	0.17 aB			11.0 aA	12.6 bA			0.60 aA	0.73 abA
Pt	0.13 bA	0.18 aA			11.0 aA	12.2 bA			0.60 aB	1.00 aA
MF	0.17		1.25		11.2		5.83		0.60	
Ct	0.10▲		1.30		12.5		6.35		0.57	

Distinct lowercase letters indicate a difference between SS-OMF physical forms and distinct capital letters indicate a difference between SS-OMF application rates by Tukey's test ($p < 0.05$). SS-OMF mean accompanied by ° differs from additional treatment MF and the mean accompanied by * differs from additional treatment Ct (Dunnett, $p<0.05$). Ct means accompanied by ▲ differs from additional treatment MF (Dunnett, $p<0.05$). Mean values (n=4).

3.4.2.5 Plant development and productivity

There was no difference between mineral fertilization and the use of SS-OMFs on soybean shoot dry mass (SDM), being significantly higher at the 100% rate compared to 70%, regardless of physical forms (Fig. 4A). On the other hand, the active nodulation (AN) was strongly influenced ($p<0.05$) by the physical forms and rates of SS-OMF, with twice as much in G and Pt compared to Pw, MF, and Ct, mainly at the 100% (Fig. 4B). A lower contact area in G and Pt physical forms and the soil can contribute to promote higher AN due to the slower chemical reactions that can cause N-losses by leaching, denitrification or access to mineral-N by soybean roots compared to Pw and MF (Correa et al., 2018). An SS-OMF in Pt physical form (75% of SS) plus filter cake promoted greater soybean development and plant growth when compared to MF (Silva et al., 2020). Similar results were obtained in the maize crop (Samuel et al., 2017). In contrast, our preliminary greenhouse study evaluating the use of the same SS-OMF (4-8-8) in soybean showed that the physical forms did not affect the early development stages of soybean and nodulation (60 days after planting) in a clay Oxisol with low fertility (Rodrigues et al., 2021). The application of N-soluble forms to the soil (NO_3^- or NH_4^+) has a strong suppressive effect on soybean nodulation, reducing the number of nodules and altering root architecture, impacting plant development (Ferguson et al., 2019; McCoy et al., 2018). Amounts between 30 - 60 kg ha^{-1} of N-soluble can inhibit the symbiotic process in the roots (Zuffo et al., 2018). Here, the SS-OMF estimated amount of N total is 25 kg ha^{-1} (100% P_2O_5-rate), considered safe to avoid nodulation suppression. Moreover, The MF treatment provided about 10 kg ha^{-1} of N-soluble (Table 1), with no differences from Ct, indicating that the application of N in soybean sowing is unnecessary (Zhou et al., 2019). Also, the heavy metal content in the SS-OMF was not worrisome since the suppression of soybean nodulation was not observed in any of the SS-OMF treatments, once at high levels, heavy metals can induce oxidative stress, and reduce the activity of key metabolic enzymes, leading to production declines, which was not observed (Baig et al., 2018; Haddad et al., 2015).

The productivity was influenced by the SS-OMF physical forms, where Pt promoted the greatest productive ($p<0.05$) compared to Pw, G, and Ct, but no differences were observed in the MF treatment (Fig 4C). The productivity mean of all treatments was following the national mean (3026 kg ha^{-1}, 2021/2022 harvest) (Conab, 2023) (Fig. 4C). Our results indicate that in soybean cultivation, the physical form of SS-OMF influenced the agronomic responses of soybeans more than the application rates, with emphasis on the physical form Pt. In this physical form, it was possible to observe a greater accumulation of P, Ca, S, and B in the soybean aerial part (Table 7) adding to lower activities of the soil enzymes Ap and As (table 9) compared to Pw and G. It indicates that it's greater hardness and smaller contact area with the soil was able to promote the slow release of nutrients and organic matter and greater use by plants, as well as by the soil microbiota, which showed fewer signs of metabolic stress due to lower enzymatic activity (Baig et al., 2018; Gong et al., 2020). This behavior is corroborated by the agronomic efficiency index (Fig. 4D) which shows that the physical form Pt was more efficient than MF. On the other hand, the physical forms Pw and G were 50% less and 75% less efficient than MF, respectively (Fig. 4D). Our result is in line with Grohskopf et al (2019) which observed about 20% more AEI in maize grain yield fertilized with a poultry litter-OMF compared to MF. Higher AEI in an OMF compared to using a strictly organic and mineral source was obtained by Mumbach et al (2020), considering two experimental years in bean, wheat, maize, and wheat season.

The application of OMFs can positively affect plant productivity beyond the addition of P in the soil, but also by the input of organic acids, micronutrients, beneficial elements, and recruitment of beneficial microorganisms in the rhizosphere, all of which can substantially contribute to gains in plant production not possible with the use of only mineral sources of nutrients (Arif et al., 2020; Bonanomi et al., 2018; de Melo Benites et al., 2022). For example, the use of SS-OMF can be an interesting source of Ni: here, the 100%-P_2O_5 rate provided 30 g ha^{-1} (Table 4). This amount is considered safe for soybean once rates above 120 g ha^{-1} can cause plant toxicity (Einhardt et al., 2021). Although Ni plays an important role in N metabolism, its supplementation is often neglected (Kutman et al., 2013; Levy et al., 2019; Yusuf et al., 2011). The addition of Ni in soybean seeds can benefit the BNF process due to the hydrogenase enzyme stimulation (Lavres et al., 2016). Further long-term studies are needed to better understand the effects of SS-OMF's physical forms and rates on soybean development.

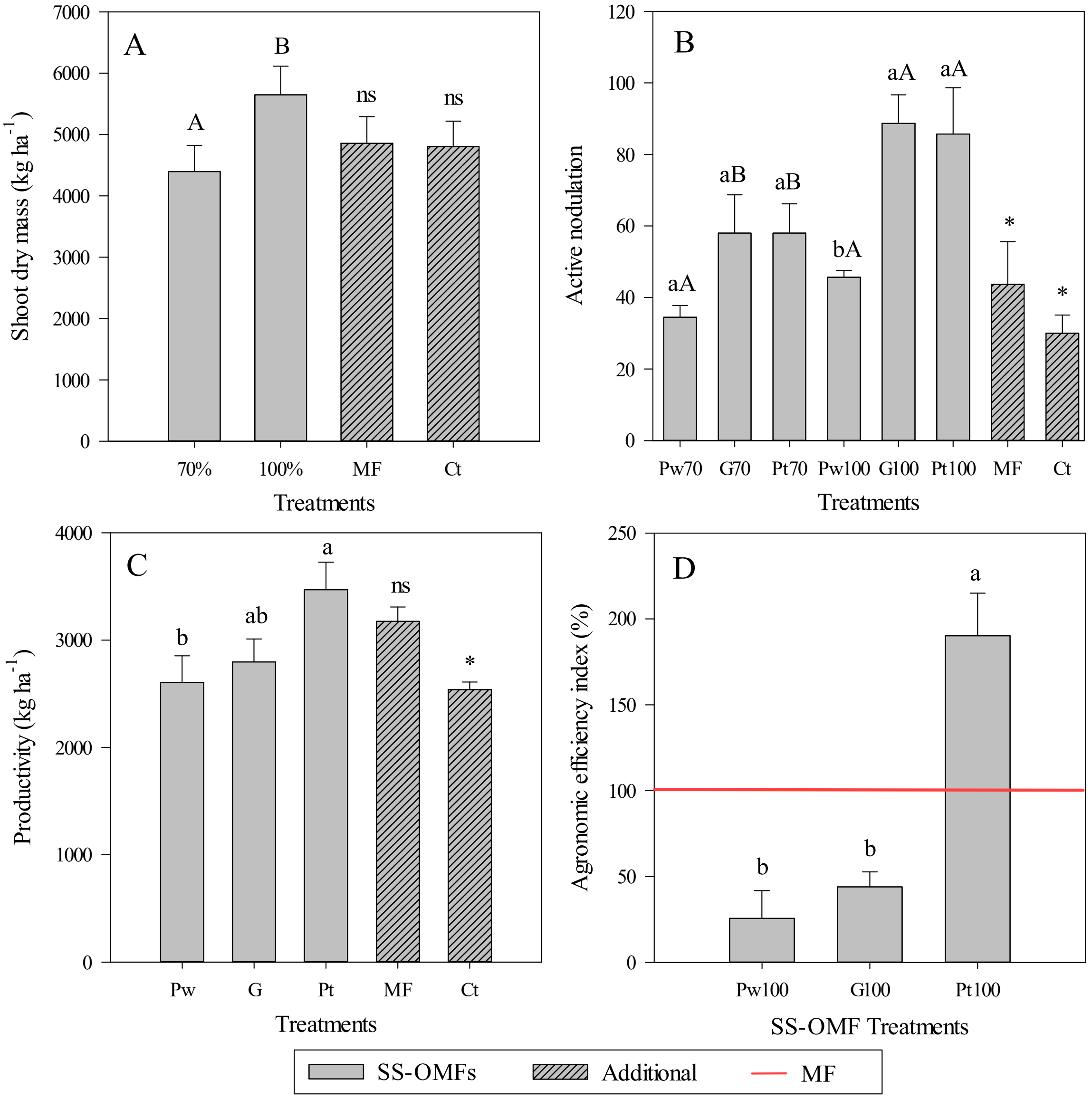

Fig. 4. Shoot dry mass (SDM) (A), active nodulation (AN) (B), productivity (C), and agronomy efficiency index (C) for soybean (*Glycine max*) amended with organomineral fertilizer from sewage sludge (SS-OMF) in tree physical forms (Pw, powder; G, granule; and Pt, pellet) and two P_2O_5 application rates (70% and 100%). Distinct lowercase letters indicate a difference between physical forms and distinct capital letters indicate a difference between application rates according to Tukey's test ($p < 0.05$). Mean values (n=4) followed by the standard error.

3.4.3 Maize off-season: residual effects

3.4.3.1 Nutritional status and nutrient accumulation

The nutritional status of residual application of SS-OMF on macro and micronutrients in the diagnosis leaves were compatible with the ranges considered adequate in maize off-season, except Mn and Zn levels, which were less than the limits of the sufficiency range in all treatments studied (Table 11) (Raij et al., 2022). However, in the field, no visual signs of deficiency of these elements were observed. There was a significant effect ($p<0.05$) of the rate of application on N content, being a 70% rate (23.1 g kg^{-1}) higher than in 100% (21.2 g kg^{-1}), however, N values were similar among SS-OMFs and MF and Ct (Table 11). Maize is highly responsive to N supply, especially in tropical soils of medium fertility and in succession with soybeans, benefiting from N remaining on the soil, requiring less at sowing (Zuffo et al., 2022).

The B content on the average of Pt (14.0 mg kg^{-1}) was higher than Ct (11 mg kg^{-}1) and MF (10.3 mg kg^{-}1) (Table 11). Regarding Fe, the average rates of Pt (113.8 mg kg^{-1}) were higher than Ct (91.6 mg kg^{-}1) and MF (92.7 mg kg^{-1}) (Table 11). This indicates greater retention of B and Fe in the pellet due to the compaction of the particles and the mixture of mineral nutrients and the organic fraction, and the consequent greater residual effect, since the B present in the SS-OMF has an organic (SS) and mineral origin (H_3BO_3) soluble, and Fe only organic origin (SS) (Fachini et al., 2021; Valentinuzzi et al., 2020). The B deficiency in plants is the most common among micronutrients in acidic soils, where its availability is favored. B losses by leaching are more pronounced compared to temperate and cold climate soils, especially in regions with high rainfall and irrigated systems, common in tropical agriculture (Padbhushan and Kumar, 2017). The low OM content in these regions contributes to the loss of B from the system since its availability in the soil is influenced by binding with SOM (soil organic matter), mainly by the aromatic groups (Schmidt et al., 2021). Also, the use of OMFs can, in addition to providing nutrients, contribute to the increase in SOM levels in the long term, reducing B leaching (G Mielki et al., 2016). Regarding the Fe content, when linked to organic components, due to the formation of soluble chelates, its solubility as well as its foliar contents in crops tends to increase (Zuo and Zhang, 2011). Moreover, the use of SS-OMF in the form of Pt favors the formation of anaerobic micro-sites, which can act in the reduction of this element, which may have influenced its better residual by maize off-season, compared to the use of Pw and G (Fachini et al., 2021; Rodrigues et al., 2021; Romano et al., 2014).

Table 11. Macro (N, P, K, Ca, Mg, and S) (g kg^{-1}) and micronutrient content (B, Cu, Fe, Mn, and Zn) (mg kg^{-1}) in the maize off-season diagnosis leaf amended with an organomineral fertilizer in three physical forms [powder (Pw), pellet (Pt), and granule (G)], two P_2O_5 rates of application (70% and 100%) and two additional treatments (MF = mineral fertilizer and Ct = without fertilization).

Treatment	**N**		**P**		**K**		**Ca**		**Mg**		**S**	
	--------------------- P_2O_5-rate (%) ---------------------											
Physical form	70	100	70	100	70	100	70	100	70	100	70	100
Pw	23.1 A	21.2 B	1.89		15.4		3.00		2.47		1.22	
G												
Pt												
MF	22.7		1.80		13.7		3.50		3.00		1.30	
Ct	23.7		1.88		14.5		3.25		2.55		1.25	
Sufficiency range*	25-35		1.9-3.5		17-30		2.5-6		1.5-40		1.5-3	
Treatment	**B**		**Cu**		**Fe**		**Mn**		**Zn**			
	--------------------- P_2O_5-rate (%) ---------------------											
Physical form	70	100	70	100	70	100	70	100	70	100		
Pw	9.00 bA	9.71 cA	5.71		94.2 b		29.2		11.6			
G	13.0 aA	13.2 bA			105.8 ab							
Pt	13.5 aA	16.3 aA°*			113.7 a°*							
MF	10.3		5.75		92.7		29.2		11.7			
Ct	11.0		6.00		91.6		31.0		11.7			
Sufficiency range*	7-17		6-15		70-200		40-100		20-50			

* (Raij et al., 2022). Distinct lowercase letters indicate a difference between SS-OMF physical forms and distinct capital letters indicate a difference between SS-OMF application rate by Tukey's test ($p < 0.05$). SS-OMF mean accompanied by ° differs from additional treatment MF and the mean accompanied by * differs from additional treatment Ct (Dunnett, $p<0.05$). Ct means accompanied by ▲ differs from additional treatment MF (Dunnett, $p<0.05$). Mean values (n=4)

In general, the residual effects of accumulation of macro and micronutrients in the maize off-season are mainly associated with a higher application rate of SS-OMF (100%), with the K and Fe content being significantly ($p<0.05$) higher in SS-OMF treatments than in MF (Table 12). Even with a lower K application in soybean (50 kg ha^{-1} in 100% P_2O_5-rate compared to 60 kg ha^{-1} in MF treatment), regardless of the physical forms, the maize off-season plants accumulated more residual-K in SS-OMF at the rate of 100% (Table 12). This can be due to the slow-release behavior of SS-OMF compared to soluble mineral sources, once K leaching is one of the major obstacles in tropical crops, mainly in irrigated systems (Alfaro et al., 2017; Bouhia et al., 2022; Goulding et al., 2021). It can indicate that the application of SS-OMF can promote better K use in crops compared to conventional mineral fertilization. Our results are in line with the greenhouse study in the short term (60 days) where higher application rates of the same SS-OMF, regarding the physical forms, increased K and P accumulation in soybean plants compared to mineral fertilization (Rodrigues et al., 2023). (Netto-Ferreira et al., 2023) observed a higher K efficiency index in SS-OMFs compared to potassium sulfate, indicating that the mixture between the organic fraction can potentially minimize K losses in the soil.

Table 12. Accumulated levels of macro N, P, K, Ca, Mg, S (kg ha^{-1}) and micronutrients B, Cu, Fe, Mn, and Zn (g ha^{-1}) in maize off-season plants amended with an organomineral fertilizer in three physical forms [powder (Pw), pellet (Pt), and granule (G)], two P_2O_5 rates of application (70% and 100%) and two additional treatments (MF = mineral fertilizer and Ct = without fertilization).

Treatment	**N**		**P**		**K**		**Ca**		**Mg**		**S**	
	---------------------- P_2O_5-rate (%) ----------------------											
Physical form	70	100	70	100	70	100	70	100	70	100	70	100
Pw	234 A	195 B	17.2 B	19.5 A*	172 B	192A°*	27.6 B	33.0 A	24.4		10.9 B	12.7 A
G												
Pt												
MF	210		17.3		162		29.9		23.4		11.5	
Ct	222		15.9		149		33.7		$31.2^{\blacktriangle}$		12.2	

Treatment	**B**		**Cu**		**Fe**		**Mn**		**Zn**	
	---------------------- P_2O_5-rate (%) ----------------------									
Physical form	70	100	70	100	70	100	70	100	70	100
Pw	124		62.6 a		892 B	1089 A°*	316 a		114	
G			55.9 ab				295 ab			
Pt			40.6 b				239 b			
MF	95.2		52.9		855		296		108	
Ct	99.6		53.9		818		294		111	

Distinct lowercase letters indicate a difference between SS-OMF physical forms and distinct capital letters indicate a difference between SS-OMF application rates by Tukey's test ($p < 0.05$). SS-OMF mean accompanied by ° differs from additional treatment MF and the mean accompanied by * differs from additional treatment Ct (Dunnett, $p<0.05$). Ct means accompanied by $^{\blacktriangle}$ differs from additional treatment MF (Dunnett, $p<0.05$). Mean values (n=4)

2.4.3.2 Heavy metal content in the soil-plant system

Similar to soybean, the heavy metals content in the soil and the maize off-season grains were much lower than those considered dangerous for human and animal health, and below the levels with potential contamination of soil and water (Appendix E and Table 13). In soil, As, Cu, Hg, Ni, Mo, and Se contents were lower than quantification limits (LQs), and in the grains, only the Fe, Zn, and Mn contents were above the LQs, however, they were lower than the limits that would make their consumption harmful (Appendix E). Our results confirmed that the residual effect of SS-OMF on heavy metals content for maize off-season was negligible. Even the exclusive application of SS takes time to promote an increase in the levels of heavy metals at levels considered to be risky (Achkir et al., 2023a). Considering the dilution of these elements and lower application rates compared to SS, the use of SS-OMFs further extends the time to use in agriculture. In the experimental conditions of this study, the risk of contamination is even lower in soil and plant, confirming the safety of using SS-OMFs (Kominko et al., 2017).

The residual levels of heavy metals in the soil after the cultivation of maize off-season were not consistently influenced by the application of SS-OMF in different physical forms and application rates or the function of the additional treatments tested (Table 14). For Cd in the soil, there is a significant effect only by physical forms ($p<0.05$) being greater in G (1.12 mg kg^{-1}) compared to Pt (0.98 mg kg^{-1}) (Table 14). On the other hand, the Pb content in the soil in Pw70 (9.32 mg kg^{-1}) was significantly higher than in the MF treatment (6.67 mg kg^{-1}) (Table 14). The Zn content in the soil was higher with the use of SS-OMF at a rate of 70% (14.7 mg kg^{-1}) compared to a dose of 100% (13.5 mg kg^{-1}) and the treatment MF (12.5 mg kg^{-1}) (Table 14). The short period of this study may have contributed to the fact that contrasting differences were not observed (1 cycle) (Kowalik et al., 2022; Nogueira et al., 2013).

Furthermore, the heavy metal content in the SS-OMF is exclusively sourced from SS which, in our case, is the raw material. The availability of heavy metals is influenced by the frequency of application in the soil, the type of SS treatment, as well as a posteriori processes such as composting, and incineration, among others (Achkir et al., 2023b; Stunda-Zujeva et al., 2018; Yadav et al., 2018). In maize grains, only the Fe content was affected by the treatments, being higher in the MF treatment (25.6 mg kg^{-1}) compared to SS-OMF Pw70 (12.7 mg kg^{-1}) and Pt100 (17.0 mg kg^{-1}) (Table 14). Therefore, the physical form of the SS-OMF did not affect the residual Zn concentration in the maize off-season grains. In the soil of the experimental area, the Zn content was considered medium (0.9 mg dm^{-3}) (Raij et al., 2022) which was enough to promote similar responses between SS-OMFs and MF treatments, not only in the Zn concentration in the grains (Table 14), but also on the nutritional status (Table 11) and accumulated (Table 12) of Zn by maize plants.

Table 14. Levels of heavy metals in the soil and maize off-season grains (mg kg^{-1}) amended with an organomineral fertilizer in three physical forms [powder (Pw), pellet (Pt), and granule (G)], two P_2O_5 rates of application (70% and 100%) and two additional treatments (MF = mineral fertilizer and Ct = without fertilization).

SOIL												
Treatment	Cd		Cr		Fe		Mn		Pb		Zn	
	---------------------- P_2O_5-rate (%) ----------------------											
Physical form	70	100	70	100	70	100	70	100	70	100	70	100
Pw	1.11 ab								9.32 aA°	6.77 aB		
G	1.12 a		35.2		30927		5.82		8.27 aA	8.50 aA	14.7 A°	13.6 B
Pt	0.98 b								7.42 aA	8.02 aA		
MF	1.02		31.2		29636		5.76		6.67		12.5	
Ct	1.12		35.5		31214		4.73		8.42		13.2	

GRAINS						
Treatment	Fe		Mn		Zn	
	---------------------- P_2O_5-rate (%) ----------------------					
Physical form	70	100	70	100	70	100
Pw	12.7 bB°*	19.8 aA				
G	20.5 aA	19.5 aA	24.2		172.2	
Pt	22.0 aA	17.0 aA°				
MF	25.6		19.2		180.6	
Ct	21.1▲		20.4		177.2	

Distinct lowercase letters indicate a difference between SS-OMF physical forms and distinct capital letters indicate a difference between SS-OMF application rates by Tukey's test ($p < 0.05$). SS-OMF mean accompanied by ° differs from additional treatment MF and the mean accompanied by * differs from additional treatment Ct (Dunnett, $p<0.05$). Ct means accompanied by ▲ differs from additional treatment MF (Dunnett, $p<0.05$). Mean values (n=4).

3.4.3.3 Bioindicators of soil quality

Differently from what was observed in soybeans, the application of SS-OMF promoted significant ($p<0.05$) residual effects in all tested soil quality bioindicators (Table 15). The activity of the enzyme βG was higher in MF than in all SS-OMFs treatments (Table 15). This enzyme is synthesized by bacteria, but mainly by fungi, and is widely used as an indicator of soil quality (Adetunji et al., 2017). The addition of organic sources, conservation management practices, and a low C/N ratio of plant residues on the soil tends to increase its activity (Reardon et al., 2022). On the other hand, factors such as salinity, the presence of contaminants, and low levels of organic matter, tend to decrease enzyme activity (Chae et al., 2017). The possibility of a suppressive effect on the microbiota from possible contaminants from the SS, such as heavy metals, organic pollutants, and drugs, is small since this behavior was seen only for this enzyme among the quality bioindicators studied (Table 15). A respirometric assay using the same SS-OMF compared to the MF treatment showed that regardless of the physical form (Pw, G, and Pt) the CO_2 emission by the microbiota was higher in the SS-OMF than the MF treatment, confirming a non-deleterious effect on the microbial community in the function of the use of SS-OMF (Rodrigues et al., 2021). Possibly the addition of OM via SS-OMF promoted a negative prime effect on microorganisms associated with the synthesis of βG in the soil, which manifested itself in maize off-season due to the non-reapplication of SS-OMF (Bastida et al., 2019; Hicks et al., 2019). Another possibility is that in the MF treatment, the supply of OM is exclusively made via soybean plant residues (no-till system), which may have forced a greater synthesis of βG by the microbiota, to optimize the acquisition of C, in the face of materials with greater recalcitrance (lignin and cellulose) (Santos et al., 2022; Wang et al., 2021). New applications of SS-OMFs are needed to confirm such findings.

In contrast to what was observed in soybeans, the Ap activity in the soil was significant ($p<0.05$) lower in the MF treatment (502 mg *p*NF kg^{-1} soil $hour^{-1}$), when compared to Pw70 (697 mg *p*NF kg^{-1} soil $hour^{-1}$), G100 (742 mg *p*NF kg^{-1} soil $hour^{-1}$), and Ct (682 mg *p*NF kg^{-1} soil $hour^{-1}$) (Table 15). The SS-OMF may have promoted the gradual release of organic-P and maintained the high Ap activity in the maize off-season compared to the MF treatment, in which there was possibly greater adsorption of P in the soil since about 75 to 90% is precipitated in the form of metal-cation complexes, being quickly fixed in tropical soils with a high content of clay and oxides of Fe, Mn, and Al (Hanyabui et al., 2020; Sharma et al., 2013). On the other hand, the greater activity in Ct reveals the balance of the adapted native microbial community since there is no addition of nutrients in this treatment and there is a need for plants to promote the acquisition of this element for their development (Andreote et al., 2014). Greater Ap activity in natural soils is observed compared to soybean and sugarcane cultivation areas in the Brazilian Cerrado due to the lower content of OM on the surface soil of cultivated areas since OM is the main component used by heterotrophic microorganisms (Leite et al., 2018).

The amount of EEG was slightly higher in SS-OMF Pt physical form and in the 70% rate (3.18 mg ml^{-1}), however similar to the MF (2.98 mg ml^{-1}) and Ct (2.92 mg ml^{-1}) (Table 15). There are no studies in the literature that show the effects of the use of SS-OMFs on the EEG. However, Choudhary et al., (2021) observed that the continuous use over 21 years of farmyard manure + NPK fertilizer proved to be the best management practice in a soybean-wheat rotation system, compared to the isolated use of these sources. Better crop productivity indices and intense biological responses in the soil were seen, where EEG was about 40% and 90% higher in this treatment than in NPK and the control without fertilization, respectively. Our results suggest that the lower access to Pi forms associated with Po input, mainly in Pt, may favor EEG synthesis by AMFs.s. It is known that the addition of Pi decreases the amounts of EEG in the soil (Aparna et al., 2014). However, long-term studies are needed to verify such findings. Furthermore, the no-tillage cultivation system favors AMFs in the soil and, consequently, greater EEG synthesis, which may have contributed to the adequate maintenance of these organisms, regardless of the treatments studied (Borie et al., 2000; Matos et al., 2022). Understanding the possible effects of the use of SS-OMF on the EEG is fundamental since its acts as a C stock in the soil that facilitates the uptake of P by plants in tropical soils (Matos et al., 2022). It plays a role in the formation of macroaggregates in the soil, which acts directly in water storage, especially important during dry periods, which are common in Cerrado production areas (Mendes et al., 2021; Wu et al., 2013; Zhang et al., 2014).

The CBM showed significant ($p<0.05$) effects only due to the SS-OMF application rate (Table 15). Here, the use of SS-OMF at 70% promoted higher CBM than 100% , but similar to Ct and MF (Table 15). Therefore, both the input of OM and mineral nutrients were able to increase CBM levels in the soil, in line with previous findings (Ma et al., 2020; Ren et al., 2019; Zhao et al., 2013). Zhang et al (2014b) observed a positive correlation between CBM and EEG after four years of application of organic amendment (crop residues or manure) in maize. The authors pointed out that MBC and EEG are abundant components of soil organic matter. In our study, only one SS-OMF application was performed, and it was not enough to change the OM contents. Thus, we can expect higher SS-OMF effects on these variables following repeated SS-OMF applications.

Table 15. Effect of SS-OMF (organomineral fertilizer from sewage sludge) in tree physical forms (Pw, powder; G, granule; and Pt, pellet) and two P_2O_5 rates (70 and 100%) on β-glucosidase (βG, mg *p*NP kg^{-1} soil hour^{-1}), acid phosphatase (Ap, mg *p*NF kg^{-1} soil hour^{-1}), arylsulfatase (As, mg *p*NS kg^{-1} soil hour^{-1}), easily extractable glomalin (EEG, mg ml^{-1}) and carbon biomass microbial (CBM, mg C kg^{-1} soil) in soil after maize off-season harvest.

Treatment	**βG**		**Ap**		**As**		**EEG**		**CBM**	
	---------------------- P_2O_5-rate (%) ----------------------									
Physical form	70	100	70	100	70	100	70	100	70	100
Pw	94.4 aA°	87.5 aA*°	697 aA°	653 aA			2.76 bA	2.91 aA		
G	114.3 aA°	92.4 aA°	521 bB	742 aA°	36.7		3.14 aA	2.92 aB	155 B	203 A
Pt	100.1 aA°	97.4 aA°	558 abA	590 aA			3.18 aA	3.02 aA		
MF	163		502		41.4		2.98		210	
Ct	129 ▲		682 ▲		27.9		2.92		160	

Distinct lowercase letters indicate a difference between SS-OMF physical forms and distinct capital letters indicate a difference between SS-OMF application rates by Tukey's test ($p < 0.05$). SS-OMF mean accompanied by ° differs from additional treatment MF and the mean accompanied by * differs from additional treatment Ct (Dunnett, $p<0.05$). Ct means accompanied by ▲ differs from additional treatment MF (Dunnett, $p<0.05$). Mean values (n=4).

3.4.3.4 Soil nutrients and fertility parameters

Only the K, Mg, Mn, and Zn levels in the soil were significantly ($p<0.05$) influenced by the treatments studied, however, the SS-OMF physical forms and applications rates did not consistently affect the residual amounts of these elements after maize off-season harvest, except K (Table 16). The average K in SS-OMF G (2.81 $mmol_c\ dm^{-3}$) was higher compared to Pt (2.15 $mmol_c\ dm^{-3}$), however, K values in the MF (3.77 $mmol_c\ dm^{-3}$) were higher than in all SS-OMF treatments, except G70 (Table 16 and Appendix D). The descending order of available K was MF > Pw = G > Pt. The K content in the soil of the experimental area is considered medium (2 $mmol_c\ dm^{-3}$, table 1) (Raij et al., 2022). On average, the use of SS-OMF at a 70% rate (2.82 $mmol_c\ dm^{-3}$) allowed more available K available in the soil than the 100% rate (2.06 $mmol_c\ dm^{-3}$). However, for both sources, the productivity was similar, indicating that SS-OMFs were as efficient as MF, despite adding less K^+ to the soil (SS-OMF 100% = 50 $kg\ ha^{-1}$ K_2O; SS-OMF 70% = 35 $kg\ ha^{-1}$ K_2O). Similar results were obtained in sugarcane, where in the first year of cultivation, the exchangeable K in the 0-20 cm layer was 15% higher in MF than in an OMF treatment (Crusciol et al., 2020). We expected that a greater residual effect of K in the soil solution would be associated with Pt physical form (Fachini et al., 2021; Kasprzycka et al., 2018; Rodrigues et al., 2021). Here, this behavior was only observed for available Mg in the Pt70 treatment (Table 16).

In both crops, the P-resin content of the soil was not influenced by the treatments (Tables 10 and 16). Similar results were obtained by Antille et al (2017) where authors did not verify the effects of an SS-OMF in extractable and the index of P in soil. However, the application of SS-OMF can replenish soil P stocks as much as MF treatment. On the other hand, Nagy et al (2020) observed an increase in the levels of soluble P in the soil that received an SS-OMF granule. However, it is important to mention that 40 $t\ ha^{-1}$ of fertilizer was applied, 64 times the application rate of this study.

Among the evaluated fertility parameters, only the pH value in the maize off-season crop showed a significant effect ($p<0.05$) as a function of the physical forms of SS-OMF. Values were slightly higher in Pw (5.85) compared to Pt (5.63) in both P_2O_5-rates (Appendix D). The slight increase in pH in Pw physical form compared to Pt may be associated with greater contact of organic matter with the soil, whose mineralization tends to decrease soil pH, as well as the presence of S°, whose oxidation process is slow and acidifies the soil (Yadav et al., 2018). A similar result was obtained in a potato crop (Ferreira et al., 2022). The fertility parameters range of values were: OM = 13.5 – 14.8 g dm^{-3}, CEC = 62.8 – 75.3 $mmol_c$ dm^{-3}, V% = 73.5 – 80.7, H+Al = 13.7 – 17.7, and SB = 46.5 – 58.6 $mmol_c$ dm^{-3}.

Table 16. Available levels of macro-K, Ca, Mg, ($mmol_c\ dm^{-3}$), P and S ($mg\ dm^{-3}$) and micronutrients B, Cu, Fe, Mn and Zn, ($mg\ dm^{-3}$) in soil (0-20 cm) after maize off-season harvest amended with an organomineral fertilizer in three physical forms [powder (Pw), pellet (Pt), and granule (G)], two P_2O_5 rates of application (70% and 100%) and two additional treatments (MF = mineral fertilizer and Ct = without fertilization).

Treatment	P		K		Ca		Mg		S	
	---------------------- P_2O_5-rate (%) ----------------------									
Physical form	70	100	70	100	70	100	70	100	70	100
Pw	32.3		2.80 aA°*	1.90 bB°	36.8		14.0 bA	14.0 aA	4.95	
G			3.13 aA*	2.50 aB°*			12.7 bA	13.7 aA		
Pt			2.53 aA°*	1.77 bB°*			20.2 aA	13.7 aB		
MF	35.6		3.77		34.0		14.2		5.50	
Ct	27.7		1.52 ▲		31.0		13.5		5.00	

Treatment	B		Cu		Fe		Mn		Zn	
	---------------------- P_2O_5-rate (%) ----------------------									
Physical form	70	100	70	100	70	100	100	70	100	70
Pw	0.20		1.33		16.1		11.5 aA	9.40 aA	0.85 aA	0.88 aA
G							8.6 bA	10.1 aA	1.07 aA	0.57 aB
Pt							9.9 abA	8.90 aA	0.70 aA	0.75 aA
MF	0.22		1.25		16.7		9.08		0.68	
Ct	0.19		1.30		17.3		9.13		0.60	

Distinct lowercase letters indicate a difference between SS-OMF physical forms and distinct capital letters indicate a difference between SS-OMF application rates by Tukey's test ($p < 0.05$). SS-OMF mean accompanied by ° differs from additional treatment MF and the mean accompanied by * differs from additional treatment Ct (Dunnett, $p<0.05$). Ct means accompanied by ▲ differs from additional treatment MF (Dunnett, $p<0.05$). Mean values (n=4).

3.4.2.5 Plant development and productivity

In maize off-season, SDM values were significantly ($p<0.05$) influenced by the physical forms of SS-OMF, where Pw and G promoted greater SDM than Pt. However, no differences were observed compared to MF and Ct treatments (Fig. 5). No significant effects ($p<0.05$) were observed in productivity of maize off-season, with an average of 6262 kg ha^{-1} among SS-OMF treatments, 6161 kg ha^{-1} on MF and 5763 kg ha^{-1} on Ct, in line with the estimated national average of 5320 kg ha^{-1} for the 2021/2022 harvest (Conab, 2023). This indicates that under the experimental conditions of this study (one application of SS-OMF and soil with medium P content), no greater residual effect on plant development and productivity was observed with the use of SS-OMF compared to the MF treatment (Sakurada L et al., 2016). A similar result was observed in soil with high P content during three years of SS-OMF application in the succession system (wheat, oilseed rape, barley, beans, and forage maize crops) (Deeks et al., 2013). On the other hand, the greater residual effect of OMFs compared to MFs is associated with their application in degraded soils or long-term (Mazeika et al., 2021).

The AEI of SS-OMF treatments follows the same pattern for soybean in terms of physical forms (Fig. 4 and 5). The G and Pt showed a higher AEI compared to Pw in maize off-season (Fig. 5). Similarly, Grohskopf et al (2019) found that after tree maize crops in soil with high P content, the poultry litter-OMF was able to provide 20% greater AEI than MF. Interestingly, the physical form Pw presented lower AEI than the MF and Ct treatments, which suggests that the use of powder impaired maize productivity compared to the additional treatments (Fig. 5). It is possible to observe a slight but significant trend towards higher Fe concentration in the grains, and Pb and Zn contents in the soil (Table 14), lower βG enzyme activity (Table 15) and K level in the soil (Table 16) with use of SS-OMF Pw compared to MF treatment that could justify negative effects on the AEI (Dhanker et al., 2020; Khaliq et al., 2017; Lamastra et al., 2018). As this behavior does not occur in G and Pt, it is possible to suggest that the physical form of the SS-OMF can be a determinant of productive responses in subsequent cultivations, since morphological aspects such as surfaces area, size, and pore volume can physically influence the availability of nutrients, heavy metals, access and mineralization of the organic fraction (Allaire and Parent, 2004; Fachini et al., 2021; Rodrigues et al., 2021; Zhang et al., 2018). For example, NH_3 emissions in a granulated organic fertilizer are six times lower than in a non-granulated (Sarauskis et al., 2021). However, studies evaluating the influence of physical forms on SS-OMFs are scarce and further investigations are needed to confirm our findings.

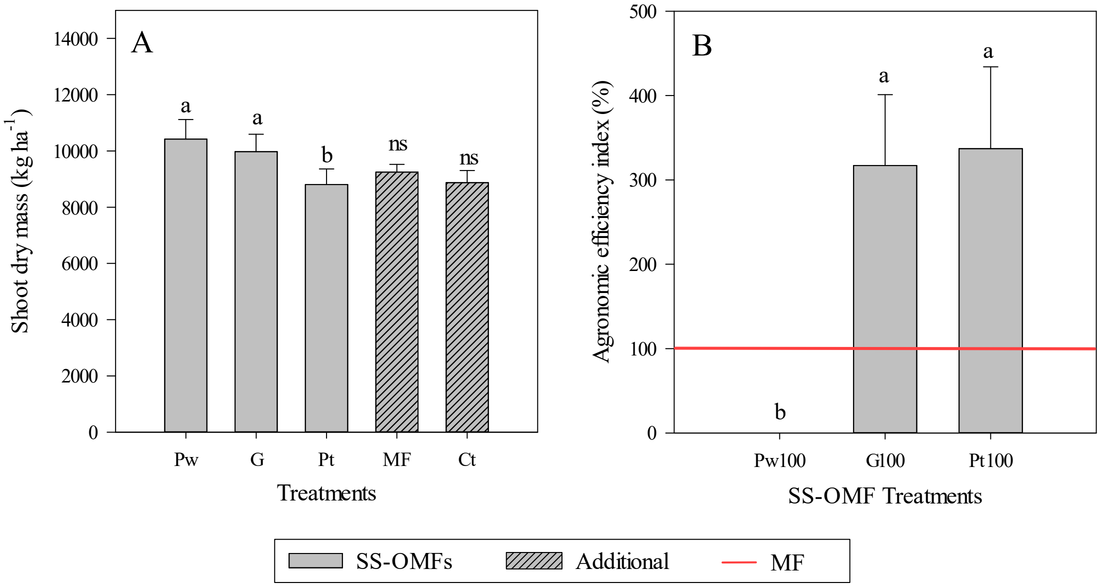

Fig. 5. Shoot dry mass (SDM) and agronomy efficiency index (B) for maize off-season (*Zea mays*) amended with organomineral fertilizer from sewage sludge (SS-OMF) in tree physical forms (Pw, powder; G, granule; and Pt, pellet) and two P_2O_5 application rates (70% and 100%). Distinct lowercase letters indicate a difference between physical forms and distinct capital letters indicate a difference between application rates according to Tukey's test ($p< 0.05$). Mean values (n=4) followed by the standard error.

3.5 Conclusions

Our results showed that sewage sludge (SS) is a safe organic matrix for the synthesis of organomineral fertilizers (SS-OMFs) and does not represent an environmental concern about the presence of sanitary contaminants and heavy metals in the soil-plant system. In an Oxisol with medium P content, the SS-OMF had equivalent effects on the soybean nutritional status, nutrient accumulation, soil fertility parameters, and shoot dry mass of plants compared to mineral fertilization (MF) regardless of the rate of application or physical form. On soybean, the levels of S and Fe in the soil and the activity of aryl sulfatase (As) enzymes were favored with the use of SS-OMF in the form of powder (Pw) while lower activity of the enzyme acid phosphatase (Ap) was obtained with the use of pellet (Pt) about MF. Soybean productivity did not differ between SS-OMFs and MF treatments, however, Pt seems to be more advantageous than Pw due to a higher agronomic efficiency index. In general, little residual effect of SS-OMF on soil and plant parameters was observed in maize off-season. However, the physical form of

SS-OMF seems to influence the availability of some nutrients and heavy metals, since the physical form Pw proved to be the least agronomically efficient for maize off-season, whose lower performance may be associated with a higher Fe concentration in grains, higher levels of Pb and Zn, and lower levels of K in the soil, and lower beta-glucosidase enzyme activity compared to MF. Thus, among the physical forms, Pt proved to be more agronomically interesting for both crops, mainly for maize off-season. The SS-OMF is a simple and environmentally safe alternative for tropical soils, allowing the proper SS disposal, recycling of OM, and nutrients. To confirm our findings, long-term field studies and economic feasibility are necessary

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APPENDIX

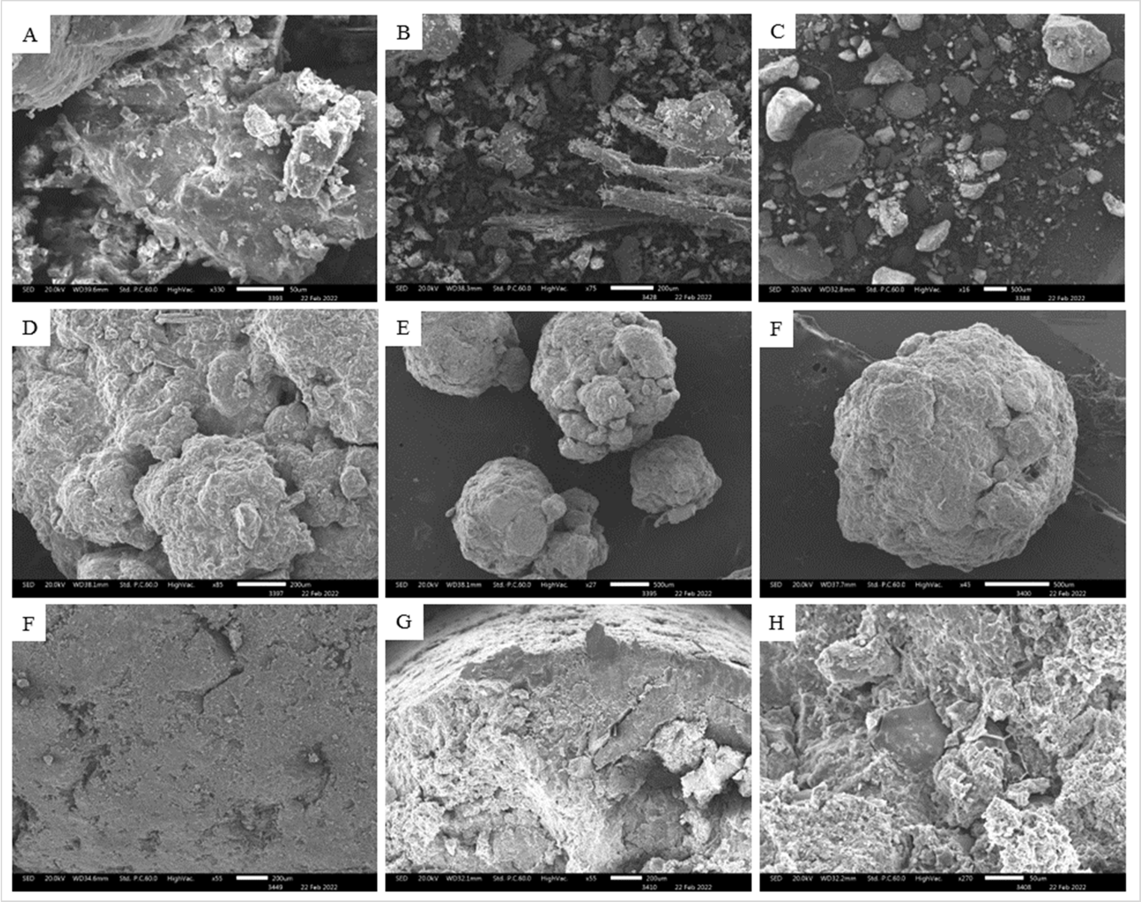
Appendix A. Granulometric distribution of organomineral fertilizer from sewage sludge in granule physical form in two masses: raw (sewage sludge + limestone without regrinding) and regrind (G) (sewage sludge + limestone).

Granule masses	**Sieve (mm)**				
	4.75	2.8	2.0	1.0	0.5
	Passing particle (%)				
Raw materials	99.23	85.65	57.37	6.21	0.97
Regrinding materials	100	81.85	57.51	13.37	2.02

Appendix B. Phytosanitary management carried out during the cultivation of soybean and maize off-season during the 2021/2022 crop year.

Growth stages	Chemical	Active ingredient	Quantity	Function
-------- Soybean --------				
V3-V4	Round Up	glyphosate	2500 g ha^{-1}	Herbicide
	Classic	chlorimuron	30 g ha^{-1}	Herbicide
R1	Perito	acephate	900 g ha^{-1}	Insecticide
	Manzate	mancozeb	1500 g ha^{-1}	Fungicide
R2-R3	Platinum Neo	thiamethoxyam + lambdacyhalothrin	200 ml ha^{-1}	Insecticide
	Privilegie	acetamiprid + pyriproxyfen	200 ml ha^{-1}	Insecticide
	Manzate	mancozeb	2000 g ha^{-1}	Fungicide
R5	Fox	trifloxystrobin + prothioconazole	4000 ml ha^{-1}	Fungicide
	Manzate		2000 g ha^{-1}	
-------- Maize off-season --------				
V1-V2	Sperto	acetamiprido	37,5 g ha^{-1}	Insecticide
	Lanate	metomil	125 ml ha^{-1}	Insecticide
V3-V4	Lanate	metomil	125 ml ha^{-1}	Insecticide
	Prêmio	clorantraniliprole	30 ml ha^{-1}	Insecticide
	Galil	imidacloprido + bifentrina	80 ml ha^{-1}	Insecticide
V7	Lanate	metomil	125 ml ha^{-1}	Insecticide
	Orthene	acefato	200 g ha^{-1}	Insecticide
	Soberan	tembotriona	48 ml ha^{-1}	Herbicide
V8	Lanate	metomil	200 ml ha^{-1}	Insecticide
	Perito	acefato	200 g ha^{-1}	Insecticide
	Galil	imidacloprido e bifentrina	80 ml ha^{-1}	Insecticide
V9	Lanate	metomil	200 ml ha^{-1}	Insecticide
	Galil	imidacloprido e bifentrina	80 ml ha^{-1}	Insecticide
R1	Lanate	metomil	200 ml ha^{-1}	Insecticide
	Perito	acefato	200 g ha^{-1}	Insecticide
R2	Lanate	metomil	200 ml ha^{-1}	Insecticide
	Prêmio	clorantraniliprole	20 ml ha^{-1}	Insecticide
	Sperto	acetamiprido	60 g ha^{-1}	Insecticide
R3	Galil	imidacloprido e bifentrina	80 ml ha^{-1}	Insecticide
	Prêmio	clorantraniliprole	20 ml ha^{-1}	Insecticide
	Lanate	metomil	200 ml ha^{-1}	Insecticide

Appendix C. Images of scanning electron microscopy (SEM) of the organomineral from sewage sludge (SS-OMF) in powder (A, 50 µm; B, 200 µm; and C, 500 µm), granule (D, 200 µm; E, 500 µm; and F, 500 µm), and pellet (G, 200 µm; H, 200 µm; and I, 50 µm) physical forms.



Appendix D. Results of the analysis of variance (F values) for the effects of the organomineral fertilizer physical forms, rates of application and additional treatments in the soybean and maize off-season crops.

Variable	Soybean						Maize off-season					
	Physical forma (PF)	Rate (R)	(PF) x (R)	Additional (A)	(A) x (PF+R)	CV (%)	Physical form (PF)	Rate (R)	(PF) x (R)	Additional (A)	(A) x (PF+R)	CV (%)
	Nutritional status (diagnose leaf)											
N	0.76	0.02	0.89	0.27	0.19	5.92	0.10	7.25*	0.28	0.71	2.53	7.43
P	0.10	0.06	0.16	0.37	0.00	6.76	0.09	0.27	0.65	0.83	1.11	6.18
K	2.86	1.87	2.86	2.42	0.04	7.74	0.90	0.16	1.58	0.50	4.24	9.86
Ca	1.03	0.29	0.03	1.01	0.00	5.90	0.63	0.84	0.21	0.63	4.26	14.4
Mg	3.02	0.32	1.23	0.01	0.80	4.88	0.30	0.80	0.05	0.44	1.33	12.7
S	0.18	0.31	0.97	0.10	1.23	7.85	0.79	1.23	0.79	0.59	2.09	7.42
B	1.32	0.00	0.60	6.98*	0.51	15.47	17.8*	2.57	1.04	0.25	5.32*	12.5
Cu	0.18	6.08*	3.01	0.61	0.26	33.98	0.84	0.30	1.28	0.07	0.09	23.1
Fe	2.60	0.43	0.50	0.18	1.47	13.08	4.83*	3.68	1.26	0.01	5.80*	12.4
Mn	0.49	0.27	0.28	0.13	0.00	12.42	1.07	0.01	3.07	0.52	0.40	11.5
Zn	0.13	0.59	2.03	1.45	0.10	23.01	0.13	3.41	0.95	0.00	0.03	9.45
	Nutrient accumulation in plant aerial part											
N	2.65	5.91*	0.63	0.17	0.56	19.23	1.40	17.2*	2.50	0.63	0.04	10.7
P	11.72*	0.73	0.21	5.05*	0.90	14.12	1.71	9.22*	1.37	1.05	5.17*	10.3
K	1.90	1.43	1.73	2.49	1.85	15.02	0.93	7.35*	2.81	1.03	13.5*	10.0
Ca	3.51*	6.33*	0.34	1.37	1.24	14.89	2.00	10.7*	2.05	1.83	0.82	13.1
Mg	2.45	5.24*	0.42	0.43	0.76	16.17	1.64	3.39	1.70	7.28*	3.09	16.3
S	3.59*	2.68	1.11	3.36	4.28	12.34	1.09	14.3*	1.44	0.69	0.05	10.0
B	3.74*	3.37	0.15	3.31	0.32	18.99	0.29	0.00	0.70	0.01	6.55	22.9
Cu	3.54*	71.18*	7.77*	0.08	0.97*	15.20	6.05*	0.00	0.09	0.01	0.00	24.4
Fe	0.11	2.50	1.21	4.94*	0.93	14.66	0.02	8.10*	0.59	0.09	4.97*	17.7
Mn	0.28	14.81*	2.85	0.28	0.00	13.71	5.43*	0.42	2.44	0.56	0.01	17.1
Zn	3.29	4.96*	2.36	1.36	0.85	18.71	1.79	4.20	0.88	0.04	0.25	16.9

Heavy metals (soil)												
Cd	1.53	0.21	0.21	1.82	0.21	22.48	4.46*	0.64	0.28	1.93	0.00	9.46
Cr	1.67	0.01	0.66	0.86	0.22	16.78	0.68	0.40	1.00	1.08	0.63	17.2
Fe	0.13	0.00	0.02	1.26	0.00	19.71	2.13	2.17	1.01	0.57	0.17	9.53
Mn	1.66	0.13	0.05	0.39	0.07	19.78	0.78	0.96	1.23	0.47	0.00	15.9
Pb	0.34	0.03	0.71	0.00	2.04	15.23	0.64	1.40	4.26*	4.48	1.09	14.7
Zn	1.94	5.57*	0.61	4.36*	3.00	12.01	0.70	4.55*	1.14	0.63	7.00*	8.64
Heavy metals (grains)												
Cu	0.54	3.23	1.13	9.82*	4.12	3.13	-	-	-	-	-	-
Fe	5.85*	0.11	1.04	0.01	5.88*	20.7	2.00	0.03	4.64*	2.54	8.34*	20.4
Mn	0.54	1.93	2.05	3.79	2.62	3.17	0.34	0.01	0.68	1.11	0.84	22.5
Zn	0.60	0.03	0.18	0.79	0.24	4.41	0.12	0.00	0.37	0.14	1.30	14.3
Bioindicators of soil quality												
βG	1.08	0.59	0.07	0.74	0.31	22.8	0.82	1.74	0.53	6.18*	37.36*	17.7
Ap	6.03*	0.13	0.29	2.31	2.90*	18.4	2.20	0.37	0.92	8.32*	0.58	15.6
As	15.5*	2.48	4.12*	4.95*	9.23*	12.5	0.79	0.37	0.32	8.32*	0.28	18.2
EEG	0.53	0.11	1.22	1.83	2.69	8.02	10.8*	2.30	5.42*	0.49	0.68	4.06
CBM	2.22	0.16	0.95	2.86	0.15	11.8	2.18	19.6*	0.23	5.23*	0.73	14.4
Soil nutrients and fertility parameters												
P	1.29	3.57	1.17	0.00	2.02	22.7	1.85	0.02	0.50	2.63	0.06	21.6
K	0.00	1.07	0.42	1.25	0.14	19.2	5.52*	20.7*	0.22	60.5*	1.62	16.4
Ca	5.68*	2.05	2.49	0.00	0.03	13.2	2.56	0.00	0.52	0.57	3.51	15.7
Mg	1.74	0.13	0.40	0.04	0.00	12.9	6.77*	4.33*	7.13*	0.24	0.98	6.77
S	11.5*	13.9*	0.65	0.85	5.18*	21.2	2.54	0.05	1.14	0.40	0.44	22.1
B	6.60*	0.31	4.00*	4.07*	2.02	30.6	0.49	0.44	1.11	1.55	0.03	18.5
Cu	1.23	0.07	0.32	0.10	0.47	16.9	0.03	0.12	0.59	0.09	2.26	8.76
Fe	5.47*	7.96*	1.57	0.63	5.03*	17.7	1.71	0.22	1.84	0.07	0.76	15.7
Mn	1.66	0.13	0.05	0.39	0.07	19.7	1.41	0.66	3.67*	0.00	1.22	13.2
Zn	2.73*	6.27*	0.99	0.03	1.03	20.1	0.86	2.56	4.11*	0.23	3.55	28.4
OM	1.72	0.02	0.32	0.55	0.74	13.5	0.55	0.00	0.70	0.05	0.12	10.4
CEC	0.74	0.01	0.21	0.00	2.27	10.3	1.76	0.24	0.17	0.47	3.61	10.4

pH	2.11	0.12	0.09	0.02	0.15	4.11	3.59*	3.62	1.88	0.04	1.61	2.78
V%	0.85	0.14	0.25	0.00	0.09	2.09	2.81	0.24	0.31	0.85	0.45	6.47
H+Al	0.26	0.03	0.03	0.00	0.00	9.73	2.85	0.52	0.25	0.70	0.04	16.2
SB	0.17	0.02	0.21	00.00	1.83	11.5	2.67	0.04	0.24	0.69	2.40	15.8
Plant development and productivity												
SDM	1.32	11.30*	0.87	0.00	0.26	18.3	3.90*	1.55	2.10	0.19	1.92	12.5
AN	12.2*	15.7*	1.25	0.25	8.06*	28.5	-	-	-	-	-	-
Productivity	9.96*	1.31	0.15	4.89*	0.36	13.9	2.28	0.04	0.49	0.18	0.80	17.2

* Significant at $p<0.05$. βG = beta-glucosidase; Ap = acid phosphatase; As = aryl sulphatase; EEG = easily exchangeable glomalin; CBM = carbon of microbial biomass; OM = organic matter; CEC = cation exchange capacity; pH = potential of hydrogen; V% = base saturation percentage; H+Al = potential acidity; SB = sum of basis; SDM = shoot dry mass; AN = active nodulation;

Appendix E. The interval of pseudo total heavy metals concentration and their prevention levels (mg kg^{-1} dry weight) in the soil (0-20 cm) and in the soybean and maize off-season grain content (mg kg^{-1} dry weight) and its respective permissible limits.

Element	Soil	(1) CETESB (2021)	Grain	(2) ABIA and (3) WHO/FAO
		Soybean		
As	<1.0	15	<1.0	NR
Cd	0.50 – 0.66	1.3	<0.2	NR
Cr	28.1 – 36.2	75	<0.6	NR
Cu	<12	60	12 - 13	30(2)
Fe	22885 – 34457	NR	100-152	214(3)
Hg	<0.5	0.5	<1.0	NR
Ni	<2.9	30	<3.2	NR
Mo	<1.3	5	<1.3	NR
Pb	5.7 – 7.0	72	<2.9	NR
Se	<1.0	1.2	<1.0	NR
Zn	6 – 8	86	42.7 – 44.0	50(2)
Mn	5.50-6.73	NR	25.3-27.0	1000(3)
		Maize off-season		
As	<1.0	15	<1.0	NR
Cd	0.99 – 1.16	1.3	<0.2	NR
Cr	31.1 – 36.2	75	<0.6	NR
Cu	<12	60	<12	30(2)
Fe	29048 - 34740	NR	17.0 – 25.6	214(3)
Hg	<1.0	0.5	<1.0	NR
Ni	<2.9	30	<3.2	NR
Mo	<1.3	5	<1.3	NR
Pb	6.7 – 9.3	72	<2.9	NR
Se	<1.0	1.2	<1.0	NR
Zn	12.6 – 14.3	86	30.1 – 33.7	50(2)
Mn	4.9-6.1	NR	18.2-25.0	1000(3)

(1) Environmental Sanitation Technology Company; (2) Brazilian Association of Food Industries; (3) World Health Organization; NR = not regulated