# RESEARCH



# Assessment of pilot-scale sewage sludge pelletization for non-food crop fertilization: nutrient content, pathogenicity, and growth performance

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Abstract Application of sewage sludge as fertilizer can be beneficial for sustainable agriculture as it could largely account for nitrogen and phosphorus demand for crops and has lower costs compared to other disposal routes, e.g., incineration, and sanitary landfills. This study evaluates the feasibility of pilotscale pelletization of sewage sludge for non-food crops (e.g., ornamental plants). The co-pelletization method was designed by mixing sewage sludge and binder (tapioca starch) at a 9:1 sludge-to-starch weight ratio. The amount of nitrogen (N), phosphorus (P), and potassium (K) of the resultant pellets were

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M. Y. Hasan Section of Bioengineering Technology, Universiti Kuala Lumpur, Kuala Lumpur, Malaysia on *Tagetes erecta* resulted in 10.5% wider leaf, 10.6% heavier leaf dry weight, and 12.5% more chlorophyll content compared to control with full usage of inorganic fertilizer. By considering liquidities to operate the production facility, the economic analysis estimated that the production cost per ton of pelletized sewage sludge produced was USD 0.98.

## Introduction

Municipal sludge, which is distinguished by its high levels of pollutants, has become an urgent worldwide environmental issue on account of its propensity to rapidly septicity if not treated. The management and elimination of municipal sewage sludge present considerable obstacles attributable to land scarcity, stringent regulatory frameworks, rising costs, and public concern. Current statistics on sewerage assets in Malaysia indicate that as of June 2019, there were over 1.3 million on-site sanitation systems in use. This includes 6839 public sewage treatment plants, 1213 network pumping stations, 3482 communal septic tanks, and 1,329,411 individual septic tanks (Hisamuddin, 2019). This accumulated to around 6 million tons of sewage sludge and suggests that this figure will rise to 10 million tons (Woo et al., 2023).

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Y. Shirai e-mail: shirai@life.kyutech.ac.jp A range of disposal techniques have been implemented, such as incineration, landfill deposition, decomposition, and natural desiccation (Farid et al., 2024). Nevertheless, recent advancements emphasize the critical nature of sustainable wastewater management, establishing it as a foundational component of Malaysia's modern sewerage infrastructure approach that emphasizes environmentally favorable technologies to alleviate the effects of climate change (Lawal et al., 2021). Prior investigations suggest that sludge that has undergone appropriate treatment possesses stable and inert properties, which make it viable for prospective applications as a soil conditioner, decomposition agent, and construction material in mining regions and for forest regeneration (IWK, 2009).

Using treated sewage sludge as a fertilizer for nonfood crops, particularly landscaping, is a novel strategy that has the potential to provide environmentally positive outcomes. Sludge that has been dewatered includes several essential nutrients, including nitrogen (N) ranging from 2.5% to 7.0%, phosphorous (P) ranging from 1% to 7.0%, potassium (K) ranging from 0.2 to 0.5%, and a variety of micronutrients that are beneficial to the development of crops (Chen & Kuo, 2016). Sludge improves soil structure by increasing porosity and water retention (Mondal et al., 2015), hence soil fertility and agricultural output over time (Börjesson & Kätterer, 2018). As shown by its favorable influence on Ailanthus altissima growth, sludge is an effective substrate for vegetation restoration (Liu et al., 2019). Abdul Khaliq et al. (2017) found that applying sewage sludge for one month increased green bean and white radish yields without causing heavy metal buildup. P. hybridum plants treated with sewage sludge had a threefold increase in production and 1.2 to 2.4 times higher soil nitrogen content than untreated plants (Lin et al., 2021). Other studies also evidenced that the sewage sludge fertilizers increased fresh biomass by 75-138% for rape, 96-138% for maize, and 23-54% for sunflower compared to control (Kominko et al., 2022). Beyond agricultural benefits, using sewage sludge as fertilizer for landscape plants reduces CO<sub>2</sub> emissions compared to municipal landfills (Chen & Kuo, 2016). With this regards, the Malaysian Government plans to fully use its market potential by 2030, particularly for land reclamation purposes (KeTTHA, 2017). The agricultural industry might enhance its efficiency and cost-effectiveness by integrating sewage sediment, which is rich in nitrogen

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and phosphate, with low-density soil additions. This approach would minimize the need for chemical fertilizers, hence benefiting socio-economic as well (Ahmad Farid et al., 2019; Hassan et al., 2024).

However, sewage sludge does have disadvantages including a low bulk, rapid deterioration, and foul odor that can be carried, transported, and stored (Puig-Arnavat et al., 2016). Due to its expanding utilization, the regulating body has enforced more stringent standards regarding the application of sewage sediment on land, necessitating either thermal drying or high-degree disinfection. Thus, the literature suggests that mechanical densification, also referred to as pelletization, is a feasible method for handling sediment while preserving its essential characteristics (Yilmaz et al., 2018). This approach addresses concerns related to transportation and application by reducing the size of the sediment, minimizing contamination, and offering cost-effective solutions (Ali et al., 2015; Chojnacka et al., 2020; Yoshizaki et al., 2013). In the early days, this technique was used to convert manure into pelletized fertilizer (Bhattacharya et al., 1989). In recent times, an increasing number of agricultural and forest by-products have been pelletized in an effort to decrease transportation and storage expenses, thereby increasing their marketability (Zafari & Hosein Kianmehr, 2012). Despite this, large-scale production has received far too little consideration.

The process of pelletizing sludge has many benefits, including improving its characteristics for different uses. Studies have demonstrated that pelletizing sewage sludge, furfural waste, and maize stalk can improve the density and mechanical characteristics of the pellets. This process also reduces the volume of sludge, making it more manageable and transportable. Additionally, it results in a more even distribution of nutrients, which can enhance plant nutrient absorption and minimize leaching into groundwater (Seleiman et al., 2020; Siyal et al., 2021). Prior to pelletization, it is essential to consider important aspects such as the concentration of heavy metals, organic micropollutants, and nutritional levels in the sludge (Koyuncu, 2022). Prior studies have investigated the use of stabilized sewage sludge as a soil fertilizer, demonstrating elevated concentrations of total nitrogen and phosphorus after its application. This contributes to improved soil nutrition and enhanced crop development. Furthermore, the use of post-floatation dairy sludge with sawdust for pelletization has been shown to reduce energy usage and enhance the quality of the pellets, rendering them appropriate for use as solid fuel (Duangjaiboon et al., 2021; Obidziński et al., 2022). These studies emphasize the importance of pelletization in improving the characteristics of sludge for advantageous use in agriculture and energy generation.

Tagetes (marigold) (Gupta et al., 2023; Kumawat et al., 2017; Sharma et al., 2017) and Celosia (cock's comb) (Abd El Gayed & Attia, 2018; Ahmed et al., 2022; Oguntade et al., 2017; Verlinden & McDonald, 2007) are species are known for their sensitivity to nutrient availability, making them excellent indicators of fertilizer efficacy. Their responsiveness allows for the detection of subtle differences in nutrient content and the subsequent impact on plant health and development. In fact, the application of sewage sludge has proven to have significant beneficial effects on the growth and productivity of marigold. Research has shown that adding sewage sludge to soil improves the development, productivity, and biochemical characteristics of marigold plants. The most successful treatment involves using 10% sewage sludge, which results in higher flower production (Al-Huqail et al., 2023). However, the use of sewage sludge in growing cock's comb is unprecedented, underscoring the novelty of our study. Thus, Tagetes erecta was selected as the control seeds for this investigation, while Celosia plumosa was also examined as an alternative ornamental plant. The physicochemical properties of the fertilizer are assessed to determine its phytotoxicity and nutrient availability. Assessment is made of the pelletized fertilizer's effect on the development of T. erecta and C. plumosa, both ornamental plants. The economic viability of the methodological approach employed in this study was also evaluated.

## Methodology

#### Materials

Sewage sludge was acquired from the Bandar Tun Razak Wastewater Treatment Plant. The sludge underwent dewatering and dehydrating procedures as its primary processes. Local markets were shopped for tapioca starch of industrial grade to use as a binder for sewage waste. Seeds of *Tagetes erecta* (Taishan® Gold) and *Celosia plumosa* (Flame<sup>TM</sup> Red) were purchased from PanAmericanSeed.

# Experimental overview

An overview of the entire project's research on the efficacy of biofertilizer pellets manufactured using a pilot-scale pelletization process for ornamental plant planting trials is depicted in Fig. 1. The pelletization process of municipal sewage sludge and binder was illustrated in the red column, while the sowing study on ornamental plants was depicted in the blue column. Extensive explanations of the experimental procedures are provided in a subsequent section.

## Pelletization of treated municipal sewage sludge

A 5.5 kW pelletizer machine (SIMA, PFH-200) with a production capacity of 200 kg h<sup>-1</sup> was utilized. Pelletizing is achieved by passing unprocessed biomass through a ring or flat die equipped with 6–8-mm apertures while three rollers apply pressure. A preliminary mixing process was conducted prior to pelletization in which the sewage sludge was uniformly combined with the following: (1) a natural binder in a 9:1 sludge to starch weight ratio and (2) water until the moisture content of the sludge reached 35 to 45% (Yilmaz et al., 2018). Subsequently, the sludge was added gradually into the hot rolling pelletizer machine.

The use of a natural binder, such as tapioca starch, in the production of sewage sludge pellets serves many essential functions. This is because the sewage sludge does not have the requisite cohesive characteristics to produce strong pellets (Li et al., 2014);



thus, a binder is needed to guarantee that the pellets stay together. Starch may function as a cohesive agent, effectively joining the sludge particles to create compacted pellets, thus avoiding their fragmentation throughout the processes of handling, storage, or transportation. Moreover, starch has the ability to absorb moisture (Zhang et al., 2024), hence preventing problems such as the softening of pellets, the growth of microorganisms, and the development of unpleasant smells during storage. Utilizing a natural binder such as tapioca starch is in line with sustainability objectives, providing an eco-friendly substitute for synthetic choices, hence decreasing the ecological consequences of manufacturing sewage sludge pellets (Karim et al., 2024; Lekniute-Kyzike et al., 2023).

Herein, the amount of starch added was adjusted to achieve the desired strength and durability of the pellets. Prior research has shown that it is recommended to use 3 wt% starch when pelletizing fertilizers made from sewage (Nikiema et al., 2013; Ofori-Amanfo, 2018). However, in our actual circumstance, it was established that at this particular ratio, the pellets that were generated were susceptible to being fractured and deformed throughout the process of pelletization. Thus, the mixing ratio is altered to provide sufficient cohesion and binding of the sludge particles, thus inhibiting the disintegration of pellets during their handling and storage (Nikiema et al., 2012). It was found that increasing the starch content to 10 wt% led to the formation of more durable pellets that were less likely to break or deform. However, a higher proportion of starch made the pellets excessively hard, which





may not be desirable for fertilizer application, which demands disintegration for its slow-release nutrients. Therefore, the 9:1 mixing ratio was selected, which gives the desirable results.

Moisture significantly influences particulate agglomeration during the pelletization process by enhancing Van der Waals interactions. However, an excessive moisture content can lead to the occlusion of mill dies. Elevated moisture levels reduce the interparticle rigidity within sludge pellets, which would otherwise form a biphasic system comprising discrete solid and liquid phases (Ungureanu et al., 2018). Consequently, moisture removal becomes imperative. The mechanical friction between the sludge and the pelletizer's die walls induces localized increases in pressure and temperature, effectively dewatering the material and causing starch gelatinization. This process enhances the adhesive interactions between the constituents, resulting in a robust composite structure. The generated friction induces viscoplastic energy dissipation, which thermally softens the binder, thereby promoting its uniform distribution within the sewage sludge matrix (Irhamna et al., 2018). Upon cooling, the pelletized sewage sludge solidifies as moisture evaporates.

Complete dehydration of the pellets is essential to inhibit microbial proliferation; thus, the pelletized sewage sludge is subjected to ambient drying for 1 h under atmospheric conditions. Nonetheless, if the pre-mixed sludge exhibits an excessively low moisture content, it may also lead to die occlusion. This is due to the heightened pelletizing pressure stemming from the vigorous frictional forces that dewater the material during pretreatment. Consequently, it is critical to maintain the moisture content of the pre-mixed sludge within an optimal range of 35 to 45%. This moisture level provides adequate lubrication within the die, reducing frictional resistance and preventing occlusion.

# Pot study setup for T. erecta and C. plumose

For this investigation, seeds of African Marigold (*T. erecta*) and cock's comb (*C. plumosa*) were considered. Given the abundance of literature regarding their phytochemistry and bioactivity, as well as their versatile applications, these plants have garnered considerable interest in scientific studies. More than that, there is minimal seedling mortality and readily

transplantable of these plants (Fadina et al., 2017; Mir et al., 2019; Oguntade et al., 2021).

Initially, T. erecta seedlings with two true leaves (10 days after sowing) were put in plastic pots, then C. plumosa seedlings after 14 days. The seedlings were fertilized in the main nursery of Taman Pertanian Universiti (TPU), located at 2.98° N latitude and 101.71° E longitude, using standard methods (Salamat et al., 2019). All pots were filled with a mixture of Serdang series topsoil and fertilizer (either inorganic or pelletized sewage sludge). The mixture consisted of three parts topsoil, two parts organic matter, and one part sand for each planting attempt. The treatments were randomized complete blocks (RCBs) with 15 replications. Growth of T. erecta and C. plumosa was regulated using light transmissibility. Using a sprinkler irrigation system, the plants were watered twice daily. Media supplied with 100% inorganic fertilizer (control) is referred to as T1, whereas media enhanced with 50% inorganic and 50% organic fertilizer is designated T2. The application of an inorganic fertilizer known as NPK Yellow (15:15:6). Fertilization was carried out weekly, starting from 2<sup>nd</sup> week, with application rates of 4.26 g per plant. The rates were increased to 12.86 g per plant in 4<sup>th</sup> week, 20 g per plant in 7<sup>th</sup> week, and 11.43 g per plant in 8<sup>th</sup> week.

As indicated in Fig. 2A, *T. erecta* and *C. plumosa* seeds were sown in the seed tray at 1 to 2 cm soil depth and covered with peat. Afterward, Fig. 2B shows the plants being moved into the pot with topsoil, organic matter, and sand after the real leaf was spotted. The plants were fertilized at 80 kg N ha<sup>-1</sup> (Król, 2012). Statistical Analysis System (SAS) version 9.2 software was then used to examine plant and soil physical appearances and nutrient availability.

#### Physicochemical characterization and phytotoxicity

Pooling and homogenizing pellet samples from each pelletization batch was performed and subsequently air-dried before analysis. Pathogenicity, dispersion, and proximate analysis were performed on 500 g of each sample. Moisture contents of raw sewage sludge and tapioca starch and produced pellets were analyzed using the A&D Moisture Analyser (MX-50, USA). Thermogravimetric analysis (TGA) was carried out using the Perkin Elmer Thermogravimetric Analyser (TGA 4000) at a continuous flow of N<sub>2</sub> at



Fig. 2 A Ten days of sowing for *T. erecta* seedlings and 14 days of sowing for *C. plumosa* seedlings are ready to be transplanted. B Seedlings were transplanted into plastic pots

10 °C min<sup>-1</sup> heating rate until 450 °C. Differential scanning calorimetry (DSC) analysis was performed using Water-LLC Differential Scanning Calorimetry (TA Instruments Q20), where samples were heated from 40 to 300 °C at a heating rate of 10 °C min<sup>-1</sup> and subsequently cooled down to 60 °C with a cooling rate of 10 °C min<sup>-1</sup>. Carbon, hydrogen, and nitrogen contents were determined by the CHNS/O Analyzer (LECO CHNS932, USA). Inductively Coupled Plasma (ICP)-OES (Perkin Elmer, USA) was used to analyze macro and micronutrients, trace elements, and heavy metals (e.g., arsenic, cadmium, cobalt, chromium, molybdenum, nickel, lead, and selenium) based on the ash obtained from the digestion method. Analysis of pathogenic microorganisms (total plate count, coliform, Escherichia coli, Staphylococcus aureus, Salmonella spp., Bacillus cereus, yeast, and mold) was carried out according to the Bacteriological Analytical Manual of Standard Method by the U.S. Food and Drug Administration (FDA BAM, 1998).

# Economic analysis

The pelletization of treated sewage requires capital investment (CAPEX) and pellet production expenditure (OPEX). The estimated cost may differ by up to 40% from the actual cost. Engineering economic principles were used to estimate expenses using equipment costs and economic multiplier values (Peters et al., 2003). Chemical Engineering Plant Cost Index and Marshall and Swift Index price cost indexes adjusted prices to current value.

## **Results and discussion**

Characteristics of sewage sludge and tapioca starch

A comparative analysis of tapioca starch and sewage sludge reveals noteworthy distinctions in their characteristics and prospective uses, as shown in Table 1. The resultant fertilizer demonstrates a harmonious equilibrium between nutrient composition and moisture content, incorporating qualities from both constituents. Sewage sludge is a moisture-rich substance; saturated sewage sludge retains approximately 5.56% of the moisture it initially contained (84.52%). The sewage sludge also demonstrates higher amounts of organic carbon (30.45%) and total carbon (40.55%) in comparison to tapioca starch (37.26% total carbon and 28.02% organic carbon, respectively). Then, 7.3% total nitrogen (TN), 3.9% total phosphorus (TP), and 0.3% total potassium (TK) are the primary nutrients most abundant in sewage sludge. Along with these macronutrients measured 2.1% calcium (Ca), 0.5% magnesium (Mg), 642.5 ppm sodium (Na), and 1.9% sulphur (S). On the contrary, the nitrogen content of tapioca starch remains undetermined, indicating the absence of its content. In addition, the sewage sludge comprises a multitude of trace elements, including manganese, iron, copper, zinc, boron, and aluminum, all of which are present in significantly higher concentrations.

The significant presence of nutrients, notably nitrogen, phosphorus, potassium, and organic carbon, in sewage sludge renders it highly promising to produce organic fertilizer. Consistent with previous studies, sewage sludge contains organic matter (ranging from

Table 1 Properties and characteristics of sewage sludge and tapioca starch

Samples	Sewage sludge	Tapioca starch	MS 1517:2012 <sup>a</sup>	US EPA 1994 <sup>b</sup>
Moisture (%)	Wet: $84.52 \pm 0.96$	$3.31 \pm 0.09$	<30	30–60
	Dried: $5.56 \pm 0.08$			
Major elements				
Total carbon, TC (%)	$40.55 \pm 1.56$	$37.26 \pm 0.10$	-	-
Organic carbon, OC (%)	$30.45 \pm 1.15$	$28.02 \pm 0.08$	-	-
Total nitrogen, TN (%)	$7.31 \pm 0.07$	nd	>1.5	-
C/N ratio	$5.58 \pm 0.25$	nd	<25	-
Total phosphorus, TP (%)	$3.90 \pm 0.02$	0.02	-	-
Total potassium, TK (%)	$0.30 \pm 0.01$	0.02	-	-
Calcium, Ca (%)	$2.09 \pm 0.03$	0.03	-	-
Magnesium, Mg (%)	$0.47 \pm 0.01$	nd	-	-
Sodium, Na (ppm)	$642.46 \pm 3.90$	nd	-	-
Sulphur, S (%)	$1.90 \pm 0.10$	$0.01 \pm 0.01$	-	-
Trace elements				
Manganese, Mn (ppm)	$160.71 \pm 3.26$	$0.85 \pm 0.07$	-	-
Iron, Fe (ppm)	$19,681.55 \pm 230.98$	$0.70 \pm 0.14$	-	-
Copper, Cu (ppm)	$89.68 \pm 5.14$	$0.35 \pm 0.07$	-	<1500
Zinc, Zn (ppm)	$798.47 \pm 22.67$	$2.85 \pm 0.49$	-	<2800
Boron, B (ppm)	$28.58 \pm 0.83$	$2.10 \pm 0.14$	-	-
Aluminum, Al (ppm)	$5133.13 \pm 86.35$	$5.40 \pm 0.28$	-	-
Heavy metals				
Arsenic, As (ppm)	$10.75 \pm 0.13$	nd	< 50	<41
Cadmium, Cd (ppm)	$1.51 \pm 0.02$	nd	<5	<39
Cobalt, Co (ppm)	$1.70 \pm 0.07$	nd	-	-
Chromium, Cr (ppm)	$18.26 \pm 2.36$	nd	< 200	-
Molybdenum, Mo (ppm)	$4.78 \pm 0.07$	nd	-	<75
Nickel, Ni (ppm)	$14.99 \pm 1.29$	nd	<150	<420
Lead, Pb (ppm)	$19.89 \pm 0.57$	$2.80 \pm 0.01$	< 300	< 300
Selenium, Se (ppm)	nd	nd	-	<100

nd Not determined.

All percentages in dry weight.

<sup>a</sup>Malaysian Standards for organic fertilizers specifications (SPAN, 2019).

<sup>b</sup>US Standards for the use or disposal of sewage sludge (USEPA, 1994).

25 to 50 wt.%) that has been shown to improve soil structure, water holding capacity, and nutrient availability for plants (Hechmi et al., 2020). Additionally, sewage sludge also comprises substantial amounts of nitrogen (2.4 to 9 wt%), primarily in the forms of proteins and amino acids (70 to 90%), which are beneficial for plant growth (Kominko et al., 2022; Usman et al., 2012). Phosphorus, another vital plant nutrient present in sewage sludge (2.3 wt%), can contribute to root growth, flowering, fruiting, and seed production (Sichler et al., 2022). Furthermore, with potassium available for grab in the sewage sludge, it may provide plants with essential elements such as potassium (0.69 to 1.03 wt%) (V. Singh et al., 2022), calcium (5.8 wt%) (Boumalek et al., 2019), magnesium (0.6 wt%) (Dusza et al., 2009), sulphur (0.3 to 2.3 wt%) (Dewil et al., 2008), as well as various micronutrients like iron (3.53 ppm), zinc (282.94 ppm), and copper (111.28 ppm) (Milik et al., 2017), which play crucial roles in metabolism (i.e., photosynthesis, respiration, and nutrient uptake), enzyme activities, and physiological processes (Özyazıcı, 2013). This characteristic positions our treated sewage sludge as a valuable substitute or supplement for agricultural crops, offering potential benefits to soil fertility and crop productivity (P. Singh et al., 2023).

Taking part in metabolic processes and serving as micronutrients, essential heavy metals, and metalloids may be crucial to a plant's growth and development at appropriate concentrations. Their effects, however, are deemed hazardous to plant growth when they are present in quantities beyond their threshold levels, especially cadmium, chromium, mercury, and lead (Wojciula et al., 2021). Heavy metals can harm plants through various mechanisms, including oxidative stress, metabolic disruptions, inhibition of growth and development, nutrient imbalances, membrane damage, and genetic alterations (Nyiramigisha et al. 2021; Pande et al., 2022; Yadav, 2010). Due to this reason, the safe utilization and disposal of our sewage sludge are called into question due to the presence of substantial concentrations of heavy metals such as arsenic (10.75 ppm), cadmium (1.51 ppm), cobalt (1.7 ppm), chromium (18.26 ppm), molybdenum (4.78 ppm), nickel (14.99 ppm), lead (19.89 ppm), and selenium (nd). Nevertheless, all remained within the acceptable range of limits established by Malaysian (SPAN, 2019) and American Standards (USEPA, 1994). Although tapioca starch is deficient in substantial amounts of nutrients, it also demonstrates negligible levels of heavy metals.

Given the safety of treated sewage sludge, it provides a rational case for the use of treated sewage sludge for different agricultural purposes in Europe, as outlined by the European Commission (2015), encompassing soil enrichment for vegetable crops, grasslands, and farmlands specifically designated for animal husbandry. Germany serves as an example in this case, where roughly half of its sewage sludge is converted into fertilizer, with the remaining either diverted for energy recovery or disposed of in landfills (BMUV, 2023). In the US, Blaine et al. (2013) reported that 60% of treatment facilities' sewage sludge has been used to improve lawns and gardens. A similar scenario can be observed in Paraná, Brazil, where sewage sludge has been utilized in agriculture since 2007 (Bittencourt, 2018). Romanos et al. (2019) found that sewage sludge use, both frequency and dose, improved soil quality over 16 years in Spain. These studies show that sewage sludge may be used in sustainable agriculture to increase soil fertility and health.

The NPK composition of the treated sewage sludge examined in this study was 7.3–3.9–0.3. It has been suggested that flowering plants flourish with a fertilizer ratio of N, 3; P, 1–2; and K, 1–16 NPK (Fang et al., 2023; Rolaniya et al., 2017). These values represent general NPK ratios commonly utilized, given the scarcity of specific studies focusing on tailored NPK ratios for marigold and cock's comb within the current literature. Nevertheless, this emphasizes the practicality of using sewage sludge as a nutrient source in landscapes, particularly for enhancing the growth of flowering plants. This is because the sewage sludge has concentration of nutrients that closely aligns with the necessary NPK ratio for flowering plants.

The thermogravimetric (TGA) analyses and decomposition temperatures of sewage sludge and tapioca starch are illustrated in Fig. 3A and B. Dehydration, caused by water evaporation and oxidative decomposition, is expressed as a decrease in mass (Ahmad Farid et al., 2017). It was evident from the TG/DTG plot that the temperatures at which degradation occurred for each component in the pelletized sludge fall within the range of 250 to 350 °C. This indicates that neither the sludge nor the binder was thermally degraded during the pelletizing process, which typically generates heat and maintains the operational die temperature between 110 and 130 °C (Nielsen et al., 2009). In addition, the resultant pellets were thermally stable, allowing them to be dried in a stovetop oven (100 °C) or under the sun (50 to 60 °C) without risk.

The present investigation employed DSC analysis to ascertain the melting points of tapioca starch and sewage effluent. The melting points of sewage sludge and tapioca starch were 208 °C and 120 °C, respectively, as illustrated in Fig. 3C and D. The thermal stability of the pellets was ensured during pelletization at a temperature below the melting points of the sludge and binder, as confirmed by the TGA. As indicated previously, friction between the die and rollers generates and maintains heat between 110 and 130 °C during pelletization. This causes the starch to gelatinize and the lignin to dissolve, which functions as a binder to maintain the structure of the particle. According to IFST (2017), gelatinization takes place at 96 °C, whereas lignin liquefaction occurs between 127 and 193 °C. Conversely, the softening temperature plummets significantly to a minimum of 54 °C due to the water sorption facilitated by the lignin (Goring, 1965). Consequently, it can be deduced that



Fig. 3 Thermogravimetric (TGA) graphs for A sewage sludge and B tapioca starch and differential scanning calorimetry (DSC) graphs for C sewage sludge and D tapioca starch

**Fig. 4** Industrial-scale pelletization process. **A** Freshly produced pelletized fertilizer and **B** industrialscale pelletization mill machine



the heat produced during pelletization is the result of a mechanism of adhesion between the particles of sewage sediment and the binder.

Physicochemical and phytotoxicity of the produced fertilizer

Three samples of co-pelletization between tapioca starch and sewage waste were utilized to produce an estimated 600 kg of pelletized organic fertilizer. The effectively fabricated pellets are illustrated in Fig. 4. The pellets span a diameter ( $\Phi$ ) of 2 to 12 mm and a length of 1 to 1.5 cm.

Insights into the impact of the pelletization process on the nutrient content of sewage sludge are provided by comparing the elemental compositions of unprocessed and pelletized sewage sludge, as presented in Table 2. An analysis was conducted on essential elements including nitrogen, phosphorus, potassium, magnesium, and calcium, in addition to trace elements

Table 2         Elemental           compositions of pre-mixed	Elemental compositions	Pre-mixed	Pelletized sludge
and pelletized sludge	Major elements		
	Nitrogen, N (%)	$5.91 \pm 0.05$	$5.69 \pm 0.49$
	Phosphorus, P (%)	$4.94 \pm 0.10$	$4.85 \pm 0.23$
	Potassium, K (%)	$0.21 \pm 0.01$	$0.19 \pm 0.07$
	Magnesium, Mg (%)	$0.46 \pm 0.02$	$0.44 \pm 0.01$
	Calcium, Ca (%)	$2.23 \pm 0.05$	$2.17 \pm 0.11$
	Trace elements		
	Boron, B (%)	$32.73 \pm 1.54$	$30.23 \pm 6.07$
	Copper, Cu (ppm)	$98.99 \pm 1.86$	$97.10 \pm 19.94$
	Zinc, Zn (ppm)	$1187.25 \pm 10.59$	$1184.50 \pm 135.79$
ns Not stated.	Iron, Fe (ppm)	$23,105.45 \pm 761.66$	$23,039.20 \pm 1349.02$
All percentages are in dry weight.	Manganese, Mn (ppm)	$201.17 \pm 1.94$	$199.25 \pm 14.18$

such as boron, copper, zinc, iron, and manganese. It is noteworthy to mention that both pre-mixed (untreated variant) and pelletized sludge contain substantial amounts of all nutrients, with no discernible reduction occurring following pelletization. Likewise, the concentrations of trace elements, such as manganese, copper, zinc, boron, and iron, demonstrate a comparatively consistent makeup in both varieties; minor inconsistencies are confined to the error margins linked to the measurements. The findings of this research suggest that while pelletization has not altered the nutrient composition of sewage sludge, its overall impact appears to be negligible. This suggests that pelletization presents itself as a viable strategy for the management of sewage sludge, with the potential to offer advantages in terms of handling and application, all the while preserving its nutritional value.

The results presented in the table indicate that the NPK values of the pelletized sludge (5.69:4.85:0.19) are not significantly different from those of the premixed sludge (5.91:4.94:0.21). However, upon comparing the NPK values of the sewage sludge (7.3-3.9-0.3) with those of the pelletized sludge, an evident distinction in nutrient composition becomes apparent. In particular, the nitrogen and potassium levels in the pelletized sewage sludge are marginally lower than those in the sewage sludge.

The disparity between the raw and pelletized sludges in terms of nitrogen and potassium concentrations can be ascribed to several pelletization-related factors. Sewage sludge is processed during pelletization, which may include dehydrating, pulverizing, binder blending, and pellet formation. These processes may result in the redistribution or loss of nutrients. Specifically, nitrogen, which is susceptible to volatilization (Watson & Kilpatrick, 1991), may undergo some degree of depletion throughout the pelletization dehydrating process. Furthermore, potassium, as a nutrient that is soluble in water, may undergo dispersal or leakage during the processing stage (Mendes et al., 2016). Therefore, unlike nitrogen, potassium leaches at a faster rate. This is likely because potassium is present in the plant exclusively in the form of a soluble ion ( $K^+$ ).

# Microbiological pathogenic test

The microbiological tests conducted on pre-mixed and pelletized sludge are compared in Table 3. This analysis provides valuable information regarding the effectiveness of the pelletization process in mitigating the presence of pathogenic microorganisms and adhering to regulatory requirements. It is worth mentioning that the total plate count of pre-mixed sludge is considerably greater  $(1.65 \pm 0.35 \times 10^7 \text{ CFU g}^{-1})$  in comparison to pelletized sludge  $(2.17 \pm 0.12 \times 10^6 \text{ CFU g}^{-1})$ . This discrepancy suggests that pelletization may result in a decrease in the microbial load. Both pre-mixed and pelletized sludge exhibit minimal quantities of coliform and E. coli, thereby satisfying the microbial safety requirements for Class A sludge as set forth by the US EPA (USEPA, 1994).

According to Briancesco et al. (2008), the existence of organisms such as *Salmonella spp.* could

<b>Table 3</b> Microbiological           test for the presence of           pathogenic microorganisms	Parameters	Pre-mixed	Pelletized sludge	US EPA 1994, Class A <sup>a</sup>
in pre-mixed and pelletized sludge	Total plate count (CFU g <sup>-1</sup> ) Coliform (CFU g <sup>-1</sup> ) <i>E. coli</i> (CFU g <sup>-1</sup> )	$1.65 \pm 0.35 \times 10^{7}$ $2.80 \pm 1.50 \times 10^{2}$ $2.65 \pm 0.65 \times 10^{2}$	$2.17 \pm 0.12 \times 10^{6}$ < 10 < 10	ns <1000 ns
nd Not detected, ns not stated.	S. aureus (MPN g <sup>-1</sup> ) Salmonella spp. (MPN g <sup>-1</sup> )	<10 nd	<3 nd	ns <3
<sup>a</sup> U.S. Standards for the use or disposal of sewage sludge (USEPA, 1994).	Yeast & mold (CFU $g^{-1}$ ) B. cereus (CFU $g^{-1}$ )	$8.41 \pm 0.67 \times 10^5$ $8.72 \pm 0.35 \times 10^5$	$5.47 \pm 0.73 \times 10^{3}$ $0.20 \times 10^{5}$	ns ns

serve as a feasible indicator for evaluating the sterility of a stable biofertilizer and the associated health hazards it poses. According to the results, the lack or minimal existence of pathogenic microorganisms, including *S. aureus*, *Salmonella spp.*, and *B. cereus*, in both sludge forms demonstrates that the treatment procedures are effective at preventing or eradicating these pathogens. Although pre-mixed sludge contains slightly more yeast and mold than pelletized sludge, both types of sludge satisfy the microbial safety requirements for Class A sludge.

Based on the combined results, it can be concluded that pelletization is a viable option for environmentally sustainable and safe utilization in diverse contexts, including fertilizer production and soil amendment for as long they adhere to regulatory requirements. This resulted from the fact that temperatures exceeding 80 °C eradicated the majority of pathogenic microorganisms present in the sewage sludge (Furuta et al., 1980). The observed decrease in microbial load in pelletized sludge suggests that the pelletization process may have the capacity to improve microbial safety, thereby emphasizing its significance in the realm of sewage sludge management strategies.

A comparable situation was observed in the context of composting, where Briancesco et al. (2008) documented that the introduction of sewage sludge during the composting process resulted in fecal contamination of the final product. The contaminants identified included *helminth ova*, *Salmonella*, *Giardia*, *Cryptosporidium*, and *Clostridium* spores. Nevertheless, once the process transitioned to the thermophilic phase, the bacterial density inevitably declined due to the incapability of microorganisms to endure temperatures around 70 °C (Belyaeva et al., 2012). Similarly, the pathogenic microorganisms (*coliforms* and *E. coli*) were eradicated in this study due to the high frictional heat (125 to 190 °C) produced during pelletization. Furthermore, a reduction in the abundance of yeast, mold, and *B. cereus* provided further confirmation that the granules produced were decontaminated by the elevated temperature. The results obtained were consistent with those of prior research conducted by Rodríguez et al. (2012) and Bazrafshan et al. (2009).

Growth response of *T. erecta* and *C. plumosa* in enriched media

To determine how pelletized sewage sludge and inorganic fertilizer impacted the growth responses of *T. erecta* and *C. plumosa*, 8 weeks of sowing experiments were conducted. T1 control treatment consists of media enriched with 100% inorganic fertilizer, and T2 combines 50% inorganic fertilizer and 50% organic fertilizer. As illustrated in Fig. 5, the physical characteristics of *C. plumosa* grown in T2-treated media during the last week of the planting trial were more pronounced in terms of color and size compared to the T1 treatment. In contrast, *T. erecta* exhibited similar color and size development across both media.

Table 4 presents data on plant growth and dry matter production for *C. plumosa* under the two different fertilization treatments. Each value is the mean of four replications, with no significant differences observed at p > 0.05 between treatments. The results demonstrate that the T2-treated media generally enhances plant growth parameters, including height ( $63.4 \pm 2.33$  cm), chlorophyll content ( $43.3 \pm 0.20$ ), and dry weights of leaves ( $6.48 \pm 0.51$  g plant<sup>-1</sup>), stems ( $6.20 \pm 0.64$  g plant<sup>-1</sup>), and flowers ( $10.10 \pm 0.28$  g plant<sup>-1</sup>), compared to T1-treated media. These findings were consistent with the



Fig. 5 Physical appearances of A *C. plumosa* and B *T. erecta* at the final week of the planting trial (T1, 100% inorganic fertilizer, and T2, 50% inorganic fertilizer+50% sewage-based fertilizer)

Samples	Plant height	Chlorophyll	Leaf dry weight	Steam dry weight	Flower dry weight	Soil pH	
	(cm plant <sup>-1</sup> )	content (SPAD reading)	(g plant <sup>-1</sup> )	(g plant <sup>-1</sup> )	(g plant <sup>-1</sup> )	Transplant	Harvest
T1 <sup>a</sup>	50.9±3.29	$41.5 \pm 0.69$	$4.20 \pm 0.54$	$4.33 \pm 0.57$	$7.93 \pm 0.66$	4.86±0.16	$5.50 \pm 0.14$
T2 <sup>b</sup>	$63.4 \pm 2.33$	$43.3 \pm 0.20$	$6.48 \pm 0.51$	$6.20 \pm 0.64$	$10.10 \pm 0.28$	$5.37 \pm 0.04$	$5.20\pm0.12$

 Table 4
 Plant growth and dry matter production of C. plumosa planting

<sup>a</sup>100% inorganic fertilizer

<sup>b</sup>50% inorganic fertilizer:50% sewage-based fertilizer

results reported by Awang et al. (2010). The slight alteration in soil pH (T1= $4.86\pm0.16$  to  $5.50\pm0.14$ ; T2= $5.37\pm0.04$  to  $5.20\pm0.12$ ) under both treatments does not appear to negatively impact plant growth.

The results presented in Table 5 suggest that the T2 treatment supports plant growth and floral development in *T. erecta* comparably to T1 treatment. Upon harvest, there was no substantial discrepancy in the height of the plants between the two treatments (T1 and T2). However, T2 treatment had resulted in higher chlorophyll content ( $58.1 \pm 1.26$ ), greater leaf width ( $7.13 \pm 0.22$  cm), and an increased number of flowers ( $9.00 \pm 0.63$ ), indicating potential benefits of incorporating sewage-based fertilizer. This is consistent with the results reported by Ghaziani et al. (2016), where the interaction between inorganic and organic fertilizers stimulated leaf growth by increasing chlorophyll content. Similar with *C. Plumosa*, the slight changes

in soil pH under both treatments (T1= $4.86\pm0.16$  to  $5.80\pm0.06$ ; T2= $5.37\pm0.04$  to  $5.83\pm0.02$ ) do not appear to negatively impact the *T. erecta* growth.

The relationship between physical and chemical indicators on plant's growth

Both *C. plumosa* and *T. erecta* typically exhibited equivalent or slightly reduced plant height in the T2 treatment compared to the T1 treatment. Nevertheless, T2 led to a more substantial increase in leaf width in *T. erecta*. This implies that while the fertilizer mix derived from sewage may not have a major impact on the plant's vertical development, it might boost the expansion of leaves. This enhancement has the potential to improve photosynthetic efficiency and overall plant strength. The chlorophyll content, quantified by SPAD measurements, consistently exhibited

Samples	Plant height (cm plant <sup>-1</sup> )	Leaf width (cm plant <sup>-1</sup> )	Chlorophyll content (SPAD reading)	Leaf dry weight (g plant <sup>-1</sup> )	Steam dry weight (g plant <sup>-1</sup> )	Flower dry weight (g plant <sup>-1</sup> )	Total no. of flowers (plant <sup>-1</sup> )	Diameter of pri- mary flower (cm plant <sup>-1</sup> )	Soil pH Transplant Harvest
Γ1 <sup>a</sup> Γ2 <sup>b</sup>	$48.5 \pm 0.69$ $46.8 \pm 1.64$	$6.38 \pm 0.34$ 7.13 $\pm 0.22$	$50.8 \pm 0.35$ $58.1 \pm 1.26$	$5.25 \pm 0.15$ $5.87 \pm 0.24$	$4.62 \pm 0.13$ $4.52 \pm 0.09$	$4.54 \pm 0.45$ $4.36 \pm 0.28$	$7.00 \pm 0.85$ $9.00 \pm 0.63$	$6.23 \pm 0.51$ $6.25 \pm 0.86$	$4.86 \pm 0.16  5.80 \pm 0.06$ $5.37 \pm 0.04  5.83 \pm 0.02$
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**Fable 5** Plant growth, dry matter production, and floral development of *T. erecta* planting

<sup>1</sup>100% inorganic fertilizer

'50% inorganic fertilizer:50% sewage-based fertilizer

greater values in T2 for both species, suggesting enhanced photosynthetic capability and a positive correlation with plant health and productivity.

Under treatment T2, both species exhibited increased leaf dry weight, indicating a greater amount of foliage biomass resulting from better nutrient availability and absorption. The disparity in stem dry weight between T1 and T2 was negligible, suggesting that the choice of fertilizer had little impact on stem biomass. Flower dry weight was greater in C. plumosa under T2, but T. erecta exhibited comparable flower dry weights across treatments, suggesting that sewage-based fertilizer may have a more pronounced positive effect on floral biomass in some species. Both species exhibited a rise in the number of flowers per plant when subjected to T2, indicating that the fertilizer mix derived from sewage improves reproductive development, possibly resulting in increased yields and improved ornamental characteristics. The diameter of the main flower exhibited no significant variation between T1 and T2 for both species, suggesting that the kind of fertilizer does not have a substantial impact on floral size.

The soil pH exhibited minor fluctuations across different treatments and time periods, although consistently maintained a sufficient range for supporting plant development. The marginal rise in soil pH under T1 and the negligible alteration under T2 indicate that the sewage-based fertilizer does not significantly modify soil acidity, hence preserving a favorable setting for nutrient absorption and plant development.

The relationship between physical and chemical indicators, such as plant height, leaf width, chlorophyll content, and soil pH, and agronomic indicators, such as the number of flowers and flower diameter, demonstrates that higher chlorophyll content and leaf dry weights in T2 indicate greater nutrient availability and absorption. This correlation is associated with enhanced photosynthetic efficiency and overall plant health. The observed rise in the number of blooms in T2 indicates that the use of sewage-based fertilizer promotes both the development of plant structures and the production of offspring, which may result in increased crop output. Sewagebased fertilizers may be used in agricultural techniques without creating significant changes in soil pH, hence maintaining soil health and nutrient availability.

Each value is a mean of four replications, which is not significantly different at p > 0.05 between treatments.

Each value is a mean of four replications, which is not significantly different at p > 0.05 between treatments.

## Economic analysis

By establishing process specifications and costs at the biorefinery scale as baseline values, an economic projection of a plant with a significant production capacity is critical when making capital-budgetary decisions (Ahmad Farid et al., 2020). The projection employed in this investigation was predicated on an

 Table 6
 Capital and operation expenditures of sewage sludge pelletization at a production capacity of 75.7 tons per year. The currency exchange rate: 1 USD=4.29 MYR

Items	Costs (thousand, USD)
Capital expenditure (CAPEX)	
Grinder with engine motor (diesel)	6.19
Pelletizer with auto switchboard	5.45
Drying area (20-tonne capacity of sewage sludge)	46.36
Electrical system	0.55
Building	2.45
Yard improvement	6.96
Total capital investment	67.97
Operation expenditure (OPEX)	
Basic labor costs <sup>a</sup>	5.35
Fringe benefits	2.14
Supervision	1.07
Administration	2.68
Operating supply	0.39
Laboratory QA/QC	1.22
Utilities <sup>b</sup>	1.89
Consumables <sup>c</sup>	39.68
Total variable costs	54.42
Depreciation <sup>d</sup>	6.46
Maintenance and repair	13.04
Total fixed costs	19.50
Total production costs	73.92
Unit cost per ton	0.98

<sup>a</sup>Calculated based on the Malaysian national minimum wage policy at USD 1.01  $h^{-1}$ 

 $^{\rm b}\text{Calculated}$  based on the amount of binder required at USD 0.7  $\rm kg^{-1}$ 

<sup>c</sup>Referred to the latest electricity tariff for the low voltage industry by Tenaga Nasional Berhad (TNB), Malaysia

<sup>d</sup>Calculated based on a 9.5-year depreciation period

annual manufacturing capacity of 75.7 tons. Capital expenditures (CAPEX) and production expenditures (OPEX) for the pelletization of sewage sludge are summarized in Table 6. The anticipated CAPEX incorporated the costs of basic materials and equipment procured at the present market value. The data presented in the table indicates that the drying area incurred the highest expenditure, constituting 68.2% or USD 46.36 thousand. The overall amount of initial capital liquidities required for the pelletization system under consideration was USD 67.97 thousand.

OPEX was determined by the amount of liquidities required for the operation and administration of the facility. To manage 330 production cycles per year on such a large scale, the facility needed to operate for eight hours per day with two operators on staff (Norrrahim et al., 2022). Under the national minimum wage policy of Malaysia, the cost of fundamental labor was USD 1.01 per hour (Ahmad Farid et al., 2021). Budgetary considerations for administrative tasks, discretionary benefits, and supervision were determined using economic multipliers. The estimated annual administrative expenses were USD 5.35 thousand or approximately 9.8% of variable costs. The total variable cost, which included wages and salaries, operating supplies, laboratory quality assurance and control, utilities, and consumables, comprised the largest proportion of production expenses at 73.6%. Depreciated asset costs and maintenance expenditures constitute the total fixed costs. Annual expenses amounting to USD 73.9 thousand were incurred by adding the fixed costs and variables, with a unit-production cost per ton of USD 0.98.

## Conclusion

The pilot-scale pelletization of sewage sludge was executed with efficacy by employing a tapioca starch to sewage sludge weight ratio of 9:1. It is worth mentioning that the nutrient content of the pellets was not substantially impacted by the pelletization procedure; the NPK composition remained consistent at 5.69:4.85:0.19. The results of the analysis confirmed that the sewage sludge's concentrations of non-essential elements complied with the regulatory limits specified in both Malaysian and US Standards. This validation affirmed the sludge's appropriateness for ornamental purposes. Notably, the pellets maintained their nutritional integrity in spite of the elevated temperatures that were encountered throughout the pelletization process. Furthermore, the pellets satisfactorily fulfilled the pathogenicity standards set forth by the USEPA. The growth performance of *T. erecta* and *C. plumosa* was found to be preferable in planting trials that utilized biofertilizer pellets, as opposed to the control group that utilized inorganic fertilizer. An estimate of USD 0.98 per ton of pelletized sewage sludge was derived from an economic analysis that accounted for annual production costs.

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