EVALUATION OF A SUBSTRATE-PLANT SYSTEM CONTAINING EFLUENT TREATMENT PLANT SLUDGE FROM A PAINT INDUSTRY

AVALIAÇÃO DE SISTEMA SUBSTRATO-PLANTA CONTENDO LODO DE ESTAÇÃO DE TRATAMENTO DE EFLUENTES PROVENIENTE DE INDÚSTRIA DE TINTAS

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ABSTRACT

The latex paint industry uses drinkable water on a large scale in its process. After used, it goes to effluent treatment plants (ETP), generating a sludge as a by-product. Currently, this residual sludge is disposed in co-processing furnaces, which creates a problem due to the high costs associated with this process This study aims to evaluate the viability of using the ETP sludge from a local latex paint industry as a substrate in the farming of Lobularia maritime. In total, five treatments composed by different amounts of sludge and substrate were performed, one being the control, containing 0% of sludge, and the others in the proportions of 10%, 20%, 30%, 40% (substitution in volume of substrate for sludge). The sludge samples and the five treatments were characterized by pH, electrical conductivity (EC), total content of soluble salts (TCSS), bulk and dry density, moisture content and particle size distribution analyses. In addition to the previous mentioned analyses, for the sludge samples, X-ray fluorescence analyses (XRF), thermogravimetric analysis (TGA/DTG) and specific mass determination were also performed, and for the treatments studied, analyses of porosity, water retention capacity and plant growth were carried out. In all treatments evaluated, the results of EC and pH were within the parameters established by the literature. The chemical analysis showed that the sludge is composed mainly of calcium (Ca), titanium (Ti), silicon (Si), aluminum (Al) and iron (Fe). As for the physical characterization, it was found that the incorporation of the residue results in an increase in density and a decrease in moisture content and in the average diameter of the system particles, negatively affecting the water retention capacity, total porosity and, possibly, the amount of micropores. All treatments showed plantular development, concluding that, when used in low proportions (up to 10% v:v), the sludge has potential use as a partial substitute material for substrate to produce seedlings.

KEY WORDS: *Substrate; ETP sludge; Latex paints; Ornamental plants; Germination*

RESUMO

A indústria de tintas látex utiliza em seu processo água potável em larga escala. Após o uso, esse insumo segue para estações de tratamento de efluentes (ETE), gerando como subproduto um lodo. Atualmente, o

descarte desse lodo residual se dá através de envio para fornos de coprocessamento, o que gera uma problemática devido aos custos elevados associados a este processo. O presente trabalho tem como objetivo avaliar a viabilidade de utilização do lodo da ETE de uma empresa de tintas látex como substrato para a produção e cultivo de Lobularia maritime. Para isso, foram realizados cinco tratamentos, compostos por diferentes quantidades de lodo e substrato, nas proporções 10%, 20%, 30%, 40% (substituição em volume de substrato por lodo) e uma testemunha, 0% de lodo. As amostras de lodo e os cinco tratamentos foram caracterizados quanto ao pH, condutividade elétrica (CE), teor total de sais solúveis (TTSS), densidade aparente e seca, teor de umidade e granulometria. Para as amostras de lodo, além das análises supracitadas, foram realizadas, ainda, as análises de fluorescência de raio X (FRX), análise termo-gravimétrica (TGA/DTG) e determinação da massa específica. Nos tratamentos estudados, foram também realizadas as análises de porosidade, capacidade de retenção de água e desenvolvimento plantular. Os resultados de condutividade elétrica e pH, encontraram-se dentro dos parâmetros estabelecidos pela literatura em todos os tratamentos avaliados. A análise química mostrou que o lodo é composto majoritariamente por cálcio (Ca), titânio (Ti), silício (Si), alumínio (Al) e ferro (Fe). Quanto à caracterização física, verificou-se que a incorporação do resíduo resulta no aumento da densidade e diminuição do teor de umidade, assim como, do diâmetro médio das partículas do sistema, afetando negativamente a capacidade de retenção de água, porosidade total e, possivelmente, a quantidade de microporos. Todos os tratamentos apresentaram desenvolvimento plantular, concluindo-se que, quando utilizado em baixas proporções (até 10% v:v), o lodo apresenta potencial uso como material substituto parcial de substrato para a produção de mudas.

PALAVRAS CHAVE: *Substrato; Lodo de ETP; tintas látex; Plantas ornamentais; Germinação.*

1. INTRODUCTION

Although the Brazilian national paint market is already consolidated, being among the five largest worldwide paint markets, it still presents the possibility of growth (ABRAFATI, 2020).

This industry uses potable water on a large scale for several purposes, among them, for cleaning operations (CETESB, 2008). According to Jewell *et al*. (2004), the washing waters represent approximately 65% of the total volume of effluents generated in the paint industry. To be reused in the process or discharged into water bodies, the effluents are treated in effluent treatment plants (ETP). As a result of the wastewater treatment process, a by-product called sludge is produced, which is a solid or pasty residue that contains impurities from wastewater (ZHANG *et al*., 2017).

According to the Brazilian legislation, solid waste must have an environmentally appropriate destination (BRASIL, 2010). Nowadays, the most commonly alternative used in latex paint industries for waste sludge disposal are landfills or co-processing furnaces. However, final disposal should be the last resource adopted, seeking, first, to reuse the waste, transforming it into a product with added value (JEWELL *et al*., 2004; RAHIMI *et al*. 2019).

Effluent treatments' sludges are considered a potential biomass for substrates, due to the high levels of organic matter present in its composition, which can bring benefits to the soil, such as density reduction, increased water retention capacity, increased total porosity, and others (BARBOSA; TAVARES FILHO; FONSECA, 2007; FERMINO, 2003). Several authors have studied the application of different types of sludge in soil as a potential agricultural substrate and the results they found were satisfactory. Farias (2018) evaluated the effects on soil fertility with the application of sewage sludgebiochar and concluded that its use resulted in greater absorption of nutrients; Sampaio *et al*., (2012) also studied the application of sewage sludge in soil, applying it in natura and aiming to evaluate its effect on the recovery of degraded areas. Santos *et al*. (2014) and Nascimento *et al*. (2020) characterized sludges of different origins in their work and evaluated the impact of their application on soil. Both concluded that the sludge is a compound rich in organic matter and important nutrients for the soil, and, taking precautions with heavy metals levels, its application is beneficial.

Although the literature on sewage sludge application on soil is vast, the same cannot be said for sludge from effluent treatment plants in the latex paint industry. Most studies related to this industry's wastewater sludge address the

possibility of incorporating this material in ceramic blocks to improve their physical and mechanical properties, mostly due to the high content of calcium and magnesium carbonate present in this residue (LODEYRO, 2011).

Due to the inputs used in the production of latex paints, it is believed that the sludge from this industry has some organic load, high mineral load, absence of heavy metals that could be harmful to the environment and desirable physicochemical characteristics for the composition of substrates. The reuse of this industrial waste as a substrate for ornamental plants can be a viable strategy from an economic, environmental, and social point of view. Therefore, the present study aims to verify the potential use of the latex paint industry wastewater sludge as a substrate in the production of seedlings for ornamental plants.

2. LITERATURE REVIEW

2.1 Paints

The paint is a homogeneous and stable mixture of resins, additives, pigments, fillers, and solvents (DINIZ, 2009). The components' proportion and distribution during the paint fabrication will be the responsible for the different types of paints in the current market (DINIZ, 2009; GOLDSCHMIDT; STREITBERGER, 2007; IKEMATSU, 2007).

The paint industry is segregated in segments according to the market the paint is intended for, being classified in i) architectural, ii) automotive, iii) automotive refinish and iv) industry inks (ABRAFATI, 2020; CETESB 2008). The most representative segment in the national market is the architectural, which, in 2019, was responsible for approximately 82.1% of the total volume of the manufactured paints (ABRAFATI, 2020; DINIZ, 2009).

Within the segment of architectural paints, the paints are again segmented according to their purpose and the base used for their manufacture, being subdivided into organic solvent-based paints and aqueous solvent paints (latex), the second being the most commercialized worldwide (DINIZ, 2009).

The manufacturing is defined according to the material used as base and it is carried out in batches, to facilitate the possible adjustments required to achieve the desired final properties (CETESB, 2008). The process follows a sequence of steps, starting at the pre-mixing and dispersing stage, going through the completion and filtration stages and ending at the paint filling and storage stage. Throughout the process, there is a constant flow of inputs and outputs of materials. The inputs are the raw materials needed for each stage, and the outputs are the waste generated (CETESB, 2008; CUSTÓDIO, 2014; LIMA, 2015; MICHALOWSKI *et al.*, 2019). In the industry in question, the environmental liabilities generated on a larger scale are effluents and solid waste.

The main source of effluents in the paint manufacturing industry comes from washing the tanks used in the process and the factory facilities, and, due to the diversity of inputs used throughout the manufacturing process, the effluent composition is quite diverse. (CUSTÓDIO, 2014; MICHALOWSKI *et al.*, 2019). The removal of the contaminants present in the effluents is carried out through physical-chemical processes (pre-treatment and primary treatment) followed by biological processes (secondary treatment). The main goal of the physical-chemical treatment is the removal of the solids and the reduction of turbidity. For this, chemical compounds are added to promote the processes of coagulation, flocculation, and sedimentation (CUSTÓDIO, 2014; GUEDES, 2018; LIMA JÚNIOR; ABREU, 2018; FRAISOLI, 2019). According to Guedes (2018), the most commonly used inorganic coagulants are aluminum and iron salts and biopolymers.

Sludge is the solid or semi-solid residue generated by the sedimentation of particles formed through the processes of coagulation and flocculation during the industrial's effluent treatment process, previously described.

The characteristics of the sludge generated by this industry vary, insignificantly, according to the ink produced and the type of product used to carry out the effluent treatment. Few studies have been found addressing this waste specifically. It is believed that, due to its origin, the sludge presents a high mineral load, from the pigments and fillers used throughout the process, and, also, some organic load content, from the products used in the effluent treatment. Depending on the inputs used in the manufacture process, the sludge may also contain traces of heavy metals in its composition (CETESB, 2008).

2.2 Plant Substrates

According to Guerrini and Trigueiro (2004), the substrates are responsible for providing the appropriate conditions for the growth, development and quality/vitality of the plants, providing better physical, chemical and biological conditions. The substrate must not only provide sufficient amounts of nutrients, water and oxygen, but also have the adequate pH and conductivity for the cultivated species and a composition free of toxic substances,

pathogens and invasive plant seeds (GUERRINI; TRIGUEIRO, 2004; LIMA *et al.*, 2006; SANTOS, 2013). Fonsêca (2001) states that any variation in the composition of the substrate can alter the seedling production process, which can cause problems from the non-germination of the seeds to the irregular development of the plants. Thus, it is important to understand the relationship that the physical-chemical characteristics of the soil culture has with the plant and root development, since these parameters vary according to the substrate used (FERMINO, 2003; TRAZZI, 2011). According to the literature, the ideal substrate must have some essential physical qualities for the proper development of the root system, as low density, high porosity, good water retention capacity, free drainage, and adequate aeration. In addition, it also must present the ideal amount of nutrients and be free from contaminants. Substrates with nutrients in excess are not recommended, since excess soluble salts can hinder plant growth (FONSÊCA, 2001; LIMA *et al.*, 2006; TRAZZI, 2011). In addition, the substrates must be economically viable, easy to obtain and have physical-chemical and morphological characteristics of little or no variation (GABIRA, 2018).

For Delarmelina *et al.* (2014), most commercial substrates formulations are low in mineral nutrients, which is essential for plant growth. To bypass this problem and improve the attributes of the substrate, the use of organic components is common. Trazzi (2011) evaluated the use of industrial and agro-industrial waste as substrates to produce forest seedlings, demonstrating that the physical and chemical properties of the substrates used are directly related to the growth of the seedlings. Silva *et al.* (2014) evaluated the growth of seedlings in treatments made up of different substrates, concluding that treatments using residues with a high content of organic matter showed high potential for use as an alternative substrate. Santos *et al.* (2014) characterized the physical-chemical properties of substrates based on sewage sludge, in different combinations, concluding that this residue had a positive impact on fertility and the amount of nutrients available in the substrates.

2.3 Effluent Treatment Plants sludge usage in agriculture

The use of sludge from effluent treatments plants (ETP) as soil substrates is considered promising from an agricultural point of view, since they have high levels of organic matter and mineral compounds in their composition, which can benefit the soil-plant system, improving the physical-chemical characteristics of the medium,

such as density, porosity, water retention capacity, among others (BARBOSA; TAVARES FILHO; FONSECA, 2007; FERMINO, 2003). The quality of the sludge is related to its generating source. Though all the sludge has a certain organic load in its composition, those generated by sewage and water treatment plants have higher levels of organic matter, while those from industrial effluent treatment plants have characteristics of the industry that originated them (FARIAS, 2018).

The disposal of sludge in soil is a frequent practice in several countries, such as the United States, China, Germany and Japan, being considered the most viable alternative, from an economic and environmental point of view, for the disposal of this waste (FARIAS, 2018; MARTINS, 2020). Currently, in Brazil, the agricultural use of sewage sludge is regulated by CONAMA Resolution 375/2006, which establishes the criteria and restrictions for this practice. The resolution limits the concentration of potentially toxic substances and defines criteria for the waste usage and the crops that can receive the disposal of the material. For Martins (2020), the reuse of this residue as a substrate in agriculture can be quite advantageous, as it reduces operating and treatment costs, increases agricultural sustainability, and improves the physical, chemical, and biological quality of the soil.

In the last decades, several studies have been carried out involving the use of different types of sludge from ETP in agriculture, aiming to evaluate the benefits that its use can offer. Delarmelina et al. (2014) studied the application of different substrates to produce Sesbania virgata seedlings and concluded that the best growth performance in height and diameter of the seedling occurred when using treatments containing sewage sludge in its composition. Santos (2013) also concluded, through his study, that the use of sewage sludge positively impacted the growth of the studied seedlings, improving the morphological and physical-chemical characteristics of the cultivation medium.

In the researches carried out by Santos *et al.* (2014) and Nascimento *et al.* (2020), sludges of different origins were characterized in order to assess the impact of their application on the soil. Both demonstrated, through their results, that, with some care regarding the existence of heavy metals, their use in agriculture is viable and beneficial to the soil, since these residues are rich in organic matter and nutrients. Sampaio *et al.* (2012) studied the effects on the physical characteristics of the soil caused by the

application of fresh sewage sludge in degraded areas, and found that the disposal of sludge resulted in improvements in the moisture content and porosity of the studied soil.

Although the results found in the literature regarding the application of sludge in the soil are satisfactory, the use of this residue in an unregulated manner can present risks to human health and to the environment, such as soil and groundwater contamination caused by the presence of metals, synthetic organic compounds, or pathogens in the waste (FARIAS, 2018; MARTINS, 2020). Thus prior to the usage of this residue as a substrate, it is extremely important to characterize it, to verify the presence of compounds in its composition that could be dangerous to the environmental.

3. METHODS

The experimental program adopted in this study was divided into three stages. More details about each stage are presented in the following subsections.

3.1 Sludge sampling and preparation

The sludge used in this research is an industrial residue from the ETP of a latex paint company located in the metropolitan region of Porto Alegre, Rio Grande do Sul, Brazil, which treats effluents from the cleaning process of both colored and white paint tanks. The sludge sampling took place according to the process described in ABNT NBR 10007: 2004.

To ensure a greater representativeness of the samples, the sampling was performed at four previously determined points of the filter press, approximately every 20 plates, and at the four ends of the plate, during a period of 5 days. The collected samples were stored in plastic bottles, at room temperature. Later, they were homogenized in the laboratory and submitted to a drying process, which was carried out in an oven for 24 h at a temperature of 105 ° C. Figure 1 shows the sludge plate formed during the process, with the ends where the sludge was collected (marked in red).

Figure 1. Sludge plate with the demarcations of the sample regions. Source: Authors.

3.2 Characterization of the samples

The characterization of the sludge samples was carried out in accordance with the instructions provided in the Normative Instruction IN SDA n° 17 of MAPA, which regulates the official analytical methods for carrying out the analysis of substrates and soil conditioners and using methodologies adapted by the institution's laboratories. The pH of the samples was determined according to IN n°17.

The pH of the samples was determined according to SDA IN n°17 of MAPA (2007). For this, the dry sample was added to a beaker with deionized water in the proportion of 1: 5 v:v. The solution was submitted to medium agitation, in an orbital shaker, for 10 minutes followed by a 15 minute rest period. After this time has elapsed, the pH was measured with the help of the TECNAL pH meter, previously calibrated. The electrical conductivity was determined to evaluate the content of water-soluble electrolytes in the substrates. The experimental procedure was the same used to determine the pH. The electrical conductivity was measured with the TECNAL conductivity meter, model Tec-4MP. The salinity of the samples was determined using the method proposed by Fermino (2003), which relates the total content of soluble salts (TCSS) present in the sample with the electrical conductivity, according to equation (1), where TCSS is the total content of soluble solids (kg / m^3), EC is the electrical conductivity (dS / m), f_c is the correction factor to express the EC in mg of KCl per liter of substrate, with a value equal to 5.6312, according to Fermino (2003) (km / dS) and ρ_a is the wet density of the sample (kg / m^3).

$$TCSS = \frac{EC \cdot f_c \cdot \rho_a}{1000} \qquad (1)$$

The moisture content was also determined using the methodology of IN SDA n° 17 of MAPA (2007). For the analysis, approximately 5 g of the sample were weighed in porcelain crucibles, which were subsequently taken to the oven at 105 ° C for 24 h. This parameter can be represented on a wet or dry basis. The moisture content on a wet basis (U_bu) is the relationship between the water mass and the total mass of the sample, as represented in equation (2), where U_bu is the moisture content on a wet basis (%), m_T is the total mass of the sample before the oven (g), m_a is the mass of water contained in the sample (g) and m_s is the mass of the dry sample (g).

121

Evaluation of a Substrate-Plant System Containing Efluent Treatment | L. K. Bickel; M. R. dos Santos; B. Brochier; I. J. Fernandes & R. C. E. Modolo

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$$\% \, U_{bu} = \frac{m_a}{m_T} \times 100 \qquad (2)$$

$$m_T = m_a + m_s \qquad (3)$$

For the determination of the apparent density (ρ_a), the visible volume is considered, which is composed of the solid part of the material and the empty spaces. The bulk density of the sludge sample was obtained by weighing an amount of dry sludge that was added to a container of known volume. The value obtained by dividing the weight of the material contained in the container by the total volume of the container is the apparent density, as shown in equation (4), where ρ_a is the apparent density, or wet density (g / mL), m is the mass of the sample added to the container (g) and V is the volume of the container (mL).

$$\rho_a = \frac{m}{V} \qquad (4)$$

The absolute density was performed using the Helium gas pycnometry method, using the Micromeritics pycnometer, model AccuPyc II 1340, from the Materials Characterization and Valorization Laboratory (LCVMat). The dry density of the sample was determined using equation (5), where ρ_s is the dry density (g / mL) and U_bu is the moisture in the sample's wet base (%).

$$\rho_s = \rho_a \frac{(100 - U_{bu})}{100} \qquad (5)$$

To determine the chemical composition and thermal decomposition of the sludge samples, X-ray and TGA/DTG and DTA fluorescence analyses were performed. For the FR-X, the Shimadzu Spectrometer model EDX-720, with detection range of 0.001% to 100%, was used and for the TGA / DTG and DTA, the simultaneous thermal analyzer of the brand Perkin Elmer, model STA 8000 with a flow of Nitrogen at a flow rate of 20 mL/min and a temperature range from 25 °C to 1000 °C, with a heating rate of 10 ° C/min was used.

3.3 Preparation, characterization, and application of the treatments

3.3.1 Preparation of the treatments

The soil used to compose the treatments of this study is from the Agroper brand, it contains only earth mixed with leaves and sticks in its composition and it is commonly used in the production of seedlings. Five treatments were adopted with different proportions of substrate (Table 1). Four treatments aimed to incorporate sludge as a substrate, in the proportions of 10%, 20%, 30% and 40% in volumetric proportion, that

is, volume: volume, and one treatment was used as a control, that is, without use of sludge.

TABLE 1. Adopted proportions for the treatments performed. Source: Authors

Treatment	Sludge (%v.v)	Soil (%v.v)
A	0	100
B	10	90
C	20	80
D	30	70
E	40	60

3.3.2 Characterization of the treatments

Analyses of salinity (electrical conductivity and total content of soluble salts), pH, apparent density (ρ_a) and dry density (ρ_s) were performed, according to methodologies described in items 3.3.1 and 3.3.3, respectively. The granulometric analysis of the treatments and the water retention capacity (WRC) were also carried out.

The granulometric analysis of the treatments took place in the same way as for the substrate, using the sequence of sieves with an aperture diameter (mm) of 4.76, 3.36, 1.68, 0.841, 0.595 and 0.42, from the series ABNT numbers: 4, 6, 12, 20, 30 and 40, respectively. From the results obtained, the average particle diameter was calculated, according to equation (6), where APD is the average particle diameter (mm), n_i is the material retained in the sieve (%) and d_i is the sieve opening diameter (mm).

$$APD = \sum_{i=1}^{n} n_i d_i \qquad (6)$$

The WRC analysis shows the maximum amount of water retained by a substrate after saturation and free drainage, without undergoing evaporation, when the tensions of 1 kPa, 5 kPa and 10 kPa are applied (BRASIL, 2007; FERMINO, 2003). The determination of the WRC was carried out by adding a predetermined amount of mass in an aluminum ring with 100 ± 5 mm internal diameter and 50 ± 1 mm in height and the bottom sealed with a screen attached with rubber ties. This system was subjected to saturation for 24 h. After this period, the cylinders were placed under the tension table for 48 h. Finally, the samples were removed and dried in an oven at 65 ° C until constant mass.

Evaluation of a Substrate-Plant System Containing Effluent Treatment | L. K. Bickel; M. R. dos Santos; B. Brochier; I. J. Fernandes & R. C. E. Modolo

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From the water retention capacity test, analyses of total porosity, microporosity and macroporosity were performed, according to the methodology proposed by Fermino (2003).

About the analysis developed, the results obtained were subjected to a variance analysis (ANOVA) test and, to verify the existence of differences among the averages, the Tukey Test was performed, with 95% of significance, using the PAST v.4.03 software.

3.3.2 Seeding and monitoring

Each treatment was arranged in a tray with 15 cells, and then sowing was carried out. Before proceeding with sowing, the materials (sludge and soil) were mixed in the desired proportions in a suitable container. Lobularia maritime, popularly known as "Flor de Mel" in Portuguese, was chosen for the study, as it is a fast-germinating plant that is tolerant of cold and frost. After determining the species and mixing the treatments, the cells in the trays were filled with the respective mixtures for each treatment. In the central part of each cell, a small hole was opened (approximately 0.5 cm^3) where about 4 to 7 seeds of Lobularia maritime were inserted. Figure 2 shows the arrangement of the trays after planting in the different treatments.
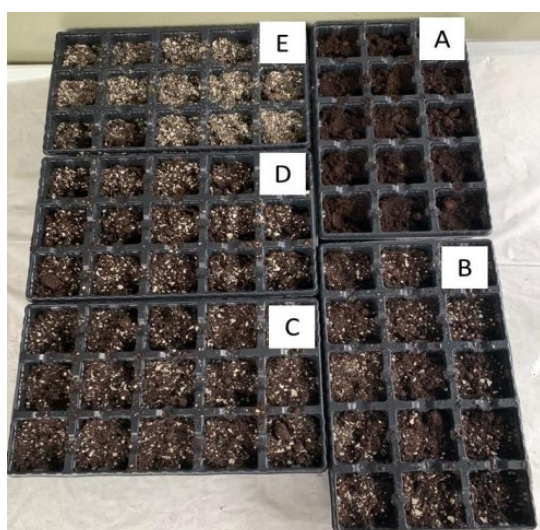

Figure 2. Trays arrangement after planting (A) soil (B) 10% sludge + 90% soil (C) 20% sludge + 80% soil (D) 30% sludge + 70% soil and (E) 40% sludge +60% soil. Source: Authors

After sowing, each cell was watered with water provided by the municipal sanitation service company, and the trays were covered with a damp cloth to keep the system moist until the germination occurred. During the experimental procedure, the trays were placed in a covered, ventilated, and well-lit place. The experiment was monitored daily for 30 days to assess seed germination and plant development. The phytometric parameters evaluated were height of the area, number of leaves and length of the roots.

Finally, the percentage of seedling survival was evaluated, according to equation (7), dividing the number of cells where the species survived (n_{cs}) by the total number of cells (n_T).

$$\%S = \frac{n_{cs}}{n_T} \qquad (7)$$

4. RESULTS AND DISCUSSION

4.1 Sludge, soil, and treatments' characterization

The results obtained for the chemical composition of the industrial waste and for the soil used are shown in Table 2. The analysis carried out for the sludge sample showed that the elements found are directly related to the pigments and fillers used in the production of latex paints. Elements such as Ca and Ti were found in more expressive amounts in the sludge composition mainly because of the compounds used in the formulation of latex paints, like dolomite [$CaMg(CO_3)_2$] and titanium dioxide [TiO_2] (SOUSA, 2019; WAN, 2013). Other products widely used in this industry are agalmatolite [$Al_2O_3(4SiO_2)(H_2O)$], talc-like materials, and kaolin [$Al_2O_3.2SiO_2$], which justifies the presence of Si and Al in significant amounts (BIZZI, SCHOBBENHAUS, VIDOTTI, 2003).

TABLE 2. A FRX results of sludge and soil samples. Source: Authors

Sample	Majority elements (>50%)	Less quantity elements (5% < x < 50%)	Trace Elements (<5%)
Sludge	-	Ca, Ti, Si, Al, Fe	Mg, K, Zr, Sr, S, Nb, Mn
Soil	-	Ca, K, Fe, Cl, P	Si, S, Mg, Cu, Zn, Mn, Al, Ti, Sr, Rb, Br

It is worth mentioning that, in the treatment of industrial effluents, the Veta L flocculant, which is composed of aluminum (Al) and iron (Fe) salts, is used in low quantities, contributing to the presence of this substances in the sludge sample. Furthermore, the levels of iron (Fe) present in the sludge can also be attributed to the pigments used to give color to the paints, since in their formulation ferric oxides are used to give red, yellow tonalities and Prussian [$Fe_4 [Fe (CN)_6]_3$] is used for bluish stains.

123

The result obtained for the soil showed that many of the elements present in the composition of the residual sludge are also present in the soil, but in different amounts, as is the case of calcium (Ca), potassium (K), iron (Fe), phosphorus (P), silicon (Si), sulfur (S), magnesium (Mg), manganese (Mn), aluminum (Al), titanium (Ti) and strontium (Sr). The elements chlorine (Cl), copper (Cu), zinc (Zn), rubidium (Rb) and bromine (Br) were found only in the soil sample.

Figure 3 shows the thermogravimetric curve of the sludge, where two main events can be observed, the first at 410.24 °C and the second at 766.93 °C, together resulting in a loss of substrate mass of approximately 41.48%. In the range of 150 to 350 °C, a slight mass loss of about 3% was noted, probably associated with the decomposition of the Prussian blue [Fe₄ [Fe (CN)₆]₃] and the organic compounds present in the sludge (AREIAS et al., 2017; SUN *et al.*, 2014; TORQUATO, 2012). Mass loss in this temperature range may also be associated with the presence of sulphates, such as aluminum, which is used as a flocculant in the treatment of wastewater and loses chemically linked water at approximately 300 °C (OLIVEIRA *et. al*, 2004).

The first thermal event with the most significant mass loss, of about 20%, was observed between 350 and 550 °C (Tpeak DTG = 410.24 °C), and the second thermal event between 625 and 825 °C (Tpeak DTG = 766.9 °C), corresponding to a mass loss of about 18.3%. According to Ávila, Crnknovic and Milioli (2010), Chiba (2017) and Areias *et al*. (2017), this loss can be attributed to the thermal decomposition of dolomite [CaMg(CO3)2], which occurs in two stages, the first being the decomposition of $MgCO_3$ and formation of MgO, in the range of 350 to 545 °C and the second, the decomposition of CaCO3 and formation of CaO, in the range of 650 to 825 °C, according to chemical reactions presented in equations (8) and (9).

$$CaMg(CO_3)_2 \xrightarrow{\Delta} CaCO_3 + MgO + CO_2 \quad (8)$$

$$CaCO_3 \xrightarrow{\Delta} CaO + CO_2 \quad (9)$$

The average values obtained for particle diameter, pH, electrical conductivity (EC), total content of soluble salts (TCSS), moisture content (Ubu), bulk density (ρ_a) and dry density (ρ_s) obtained for the samples of the sludge and the treatments are shown in Table 3.

The size of the particles has a significant influence on parameters as density, humidity and aeration of the substrate (FERMINO, 2003; SANTOS, 2013; ZORZETO *et al.*, 2014). For Zorzeto et al. (2014), particles larger than 3.35 mm are considered very large; between 3.35 and 2.00 mm are large; between 2.00 and 0.50 mm in between; between 0.50 and 0.10 mm are thin; and smaller than 0.10 mm are very thin. The different particle proportions directly influence the availability of water and air in the substrates. Very fine particles decrease the pore spaces and increase the density, decreasing the aeration of the cultivation médium.
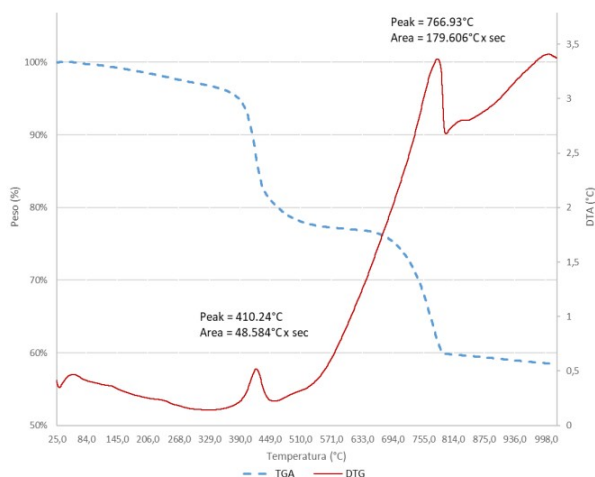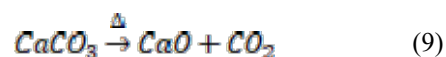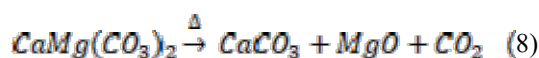
Figure 3. Thermogravimetric curve of the sludge. Source: Authors

TABLE 3. Results of particle diameter, pH, electrical conductivity (EC), Total Content of Soluble Salts (TCSS), moisture content (Ubu), bulk density (ρ_a) and dry density (ρ_s). Source: Authors. Same letters indicates that there was not a significative difference among the results for p≤0.05.

Sample	APD (mm)	pH	EC (dS.m-1 at 25°C)	TCSS (kg.m-3)	U_{bu} (%)	ρ_a (kg. m-3)	ρ_s (kg. m-3)
Sludge	2.00	$8.0^a \pm 0.10$	$0.967^a \pm 0.08$	-	$34.4^a \pm 0.6$	$784^a \pm 10$	$784^a \pm 11$
A	1.70	$6.2^b \pm 0.06$	$1.566^b \pm 0.09$	$5.05^a \pm 0.2$	$70.5^b \pm 0.3$	$573^b \pm 4$	$169^b \pm 11$
B	1.59	$6.4^c \pm 0.05$	$0.729^c \pm 0.04$	$2.74^b \pm 0.08$	$58.0^c \pm 0.8$	$668^c \pm 13$	$281^c \pm 9$
C	1.61	$6.5^c \pm 0.05$	$0.714^c \pm 0.08$	$2.72^b \pm 0.3$	$48.9^d \pm 0.2$	$677^c \pm 2$	$346^c \pm 9$
D	1.57	$6.9^d \pm 0.08$	$0.662^d \pm 0.05$	$2.64^b \pm 0.2$	$43.9^e \pm 0.7$	$709^d \pm 8$	$398^d \pm 13$

E	1.60	$7.2^e \pm 0.05$	$0.579^d \pm 0.05$	$2.47^b \pm 0.2$	$33.4^f \pm 2.3$	$760^e \pm 26$	$506^e \pm 39$

Among the samples, the sludge had the largest average particle size, of 2.0 mm. This fact may be associated with the non-total breakdown of the clods. While treatment A, composed entirely of soil, presented the smallest average particle size, of 1.7 mm. Figure 4 shows the particle size distribution of the sludge and of the five treatments studied. Treatments composed by different materials have particles with different sizes, resulting in different arrangements (LIMA, 2019).

It was observed that the increase in the amount of sludge in the treatments caused the reduction of the average particle diameter, which, in the treatments containing sludge were in the range of 1.57 to 1.61 mm. All the evaluated treatments presented varied granulometries, with predominance of very large particles, with a diameter above 3.35 mm, and intermediate particles, with a diameter between 2.00 and 0.5 mm. However, in treatments C, D and E it was noticed an increase in the percentage of particles considered intermediate and fine, with diameters between 0.5 mm and 0.10 mm.
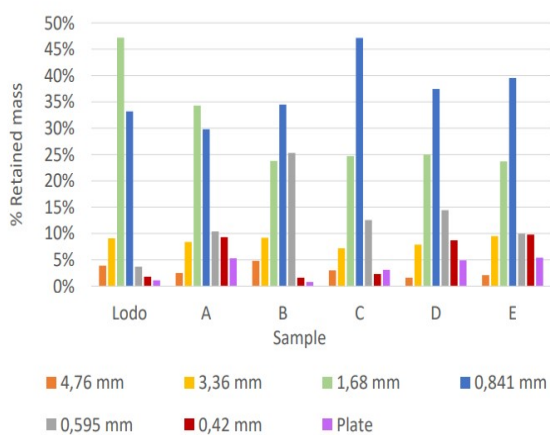

Figure 4. Particle size distribution. Source: Authors

According to Lima (2019), the physical-chemical characteristics of the particles influence properties such as the cation exchange capacity (CEC), the availability and movement of water and air in the soil, the expandability, and the susceptibility to erosion. For Zorzeto *et al.* (2014), substrates with a smaller particle size, with predominantly fine and / or intermediate fractions, have high microporosity, favor the water retention capacity and the aeration of the medium. Substrates with homogeneous particle size distribution may present a decrease in

porosity, due to the accommodation of the smaller particles between the larger particles.

It was possible to verify that the pH of the raw sludge has basic characteristic, presenting a value of 8.0 ± 0.1 at 21.2°C and that the treatment A, composed solely of earth presents a slightly acidic pH, of 6.2 ± 0.1 at 21.9°C, that is ideal for plants substrates (pH and salinity, also shown in Table 3). The samples from treatments B and C, as well as treatment A, presented slightly acidic pH and samples D and E neutral pH. It is believed that this fact is related to the composition of the treatments, since the last two treatments, which have higher sludge contents, presented higher pH values, indicating that the addition of sludge to the treatment composition causes an increase in the pH of the medium. The same was observed by Trazzi (2011), where the substrate incorporation resulted in the increase of the pH of the treatments.

The pH affects the availability of soil nutrients. For Santos (2013), the ideal pH for plants' development varies according to the species being cultivated. According to the literature, for the growth to be favorable, the pH of the substrate must be in the range of 5.5 to 6.5 (FERMINO, 2003; SANTOS, 2013). Cultivation mediums with a more acidic pH, below 5.5, indicates the presence of elements that can hamper the development of the plant, such as aluminum (Al) and manganese (Mn). Higher pH values indicate the deficiency of necessary elements in the medium, such as iron (Fe), zinc (Zi), copper (Cu), phosphorus (P) and magnesium (Mg). According to the FRX analysis carried out and previously discussed, the sludge used in the treatments studied in this research contains iron (Fe), titanium (Ti), calcium (Ca) and silicon (Si) in quantities higher than 5%, in addition to having, in amounts less than 5% magnesium (Mg) and potassium (K), which may explain its basic character.

The electrical conductivity (EC) indicates the concentration of soluble salts present in the substrate. Cultivation medium with high conductivity values can cause everything from reduced fertility and problems related to germination to negative effects on plant development, causing damage to the roots, which can prevent or hinder the absorption of water and nutrients (SCHOSSLER *et al.*, 2012). According to Cavalcante *et al.* (2002), the electrical conductivity of the substrate must not exceed 1.3 dS.m^{-1}. For Guerrini and Trigueiro (2004) and Santos (2013) the limit value of the

125

conductivity is even lower, of 1.0 dS.m⁻¹. It was except that treatment A presented EC levels within the recommended by Guerrini & Trigueiro (2004), Santos (2013) and Cavalcante *et al.* (2002). The highest and lowest EC values were observed in treatment A and E, respectively. When sludge was added to the treatments, a tendency of reduction of the EC levels was observed, indicating that this addition could decrease the EC in the cultivation medium.

The results show that the amount of sludge directly impacted the parameters of density and moisture content of the soil. When present in the mixture, the sludge was responsible for reducing the moisture content of the soil, with the highest humidity being seen in treatment A and the lowest in treatment E. This trend was also observed by Zorzeto *et al.* (2014) in their study.

For Lima (2019), the moisture content in the soil impacts the availability of minerals. Soils with a high moisture content provide the slow decomposition of organic matter, solubilizing minerals such as iron (Fe) and manganese (Mn) and facilitating their migration, being important that the material does not present high humidity, since these elements, when in excess, are toxic to the environment. To Fermino (2003), the soil moisture content is related to the arrangement of the particles in the container and the density of the medium, being directly impacted by characteristics such as the weight of the particles and the existing adhesion between them. Lower moisture content results in substrates with higher density, due to the increased weight of the particles. Intermediate

levels of humidity result in a decrease in density due to the aggregation of the particles and the formation of empty spaces and higher levels of humidity cause an increase in adhesion between the particles, generating packaging phenomena. Among the samples analyzed, fresh sludge had the highest wet and dry density, of 787 kg.m⁻³ and 784 kg.m⁻³, respectively, and treatment A had the lowest wet and dry density, of 668 kg.m⁻³ and 169 kg.m⁻³, confirming the statement of Fermino (2003).

According to Trazzi (2011), very high densities make it difficult to grow seedlings in containers, causing limitations on plant growth. Kämpf (2000) recommended using substrates with dry density between 100 and 300 kgm⁻³, for trays, between 300 and 500 kgm⁻³, for intermediate-sized pots, between 20 and 30 cm and between 500 and 800 kg m⁻³ for larger containers. That said, only treatments A and B have densities suitable for growing in trays.

4.1.1 Porosity and WRC

The quantity and quality of pores determine the distribution of solids, water and air in the substrates (GUERRINI; TRIGUEIRO, 2004). Table 4 presents the mean results obtained for porosity, EAW, BW and AS analyses. In this study, all treatments showed high porosity, above 50%. For Rigon et al. (2015) and Fermino (2003), the ideal porosity is between 75 and 85%. Thus, we realize that all treatments, except for A and B, have porosities considered ideal according to the literature.

TABLE 4. Results of Macroporosity, Microporosity, Total porosity, Easily Available Water (EAW), Buffering Watter (BW) and Aerations Space (AS). Source: Authors. * Same letters indicates that there was not a significative difference among the results for p≤0,05

Sample	Macroporosity (%)	Microporosity (%)	Total Porosity (%)	EAW (cm³.cm⁻³)	BW (cm³.cm⁻³)	AS (cm³.cm⁻³)
A	13ª ± 2,9	83ª ± 2,8	97ª ± 3,8	36ª ± 2,0	1ª ± 0	13ª ± 2,5
B	10ª ± 0,3	78ᵇ ± 1,0	88ᵇ ± 1,1	35ª ± 1,2	1ª ± 0	10ª ± 0,0
C	12ª ± 1,9	74ᵇ ± 0,9	86ᵇ ± 1,2	32ᵇ ± 1,0	0ª ± 0	12ª ± 1,5
D	11ª ± 1,9	72ᶜ ± 1,9	83ᵇ ± 1,8	34ª ± 1,7	0ª ± 0	11ª ± 2,0
E	13ª ± 2,6	62ᵈ ± 1,5	75ᶜ ± 1,2	29ᶜ ± 1,0	0ª ± 0	13ª ± 2,5

It was possible to observe a trend in the reduction of porosity as the volume of the sludge incorporated in the treatments increased. Treatment A and treatment E had the highest and the lowest total porosity values, respectively. According to Zorzeto *et al.* (2014), when there is a mixture of materials of different

particle sizes, the smaller particles tend to occupy the empty spaces between the larger particles, which reduces the amount of macro and micropores and, consequently, the total porosity.

In substrates with high amounts of sludge, a reduction in the amount of micropores was noticed, which hinders the retention and storage of water from the substrate, damaging the development of the root system (PAGLIARINI; CASTILHO; ALVES, 2012). As for the percentage of macropores in the treatments, it did not show significant variation with the increase of sludge incorporated into the treatment.

Figure 05 shows the retention curve obtained for the different treatments when submitted to different tensions, of 1, 5 and 10 kPa. All materials showed high values of water retained in the different stresses evaluated, in the range of 62 to 87 m3 m^{-3} in the analysis of CRA1, and 33 to 47 m3 m^{-3} in the analyzes of CRA5 and CRA10. For the evaluated points, it was possible to observe that the addition of sludge in the treatments caused a decrease in the water retention capacity of the samples. Treatment A, composed entirely of vegetable soil, has the largest volume of water retained, while treatment E, composed of 60% vegetable soil + 40% sludge, presented the lowest volume of water retention.
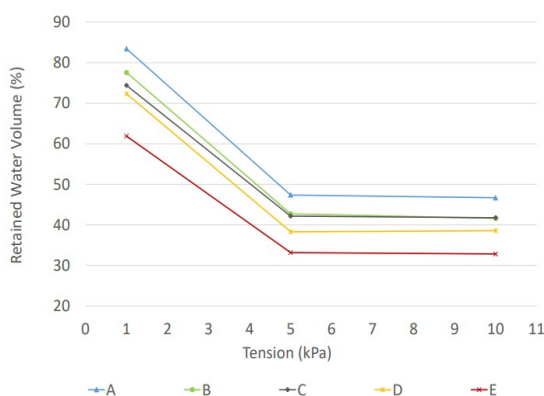

Figure 5 - Water retention curve of the different treatments subjected to voltages of 1, 5 and 10 kPa. Source: Authors.

For Guerrini e Trigueiro (2004), the water and nutrient retention capacity of the substrate is directly related to microporosity, once high microporosity values, in general, result in a greater water retention capacity of the substrate. The result observed in the present work is similar to those observed in previous studies, where the increase in the sludge content in the treatments reduced their microporosity and their total porosity and, consequently, the water retention capacity of the substrate (FERMINO, 2003; GUERRINI; TRIGUEIRO, 2004; PAGLIARINI; CASTILHO; ALVES, 2012; RIGON *et al.*, 2015; TRAZZI, 2011). Thus, the use of sludge as a substrate at levels above 10% v: v is not viable to produce seedlings, due to the high

consumption of irrigation water that would be necessary.

The treatments were also evaluated for aeration space (AS), easily available water (EAW) and buffering water (BW), as shown in Table 04. In ideal conditions, the aeration space (AS) should present between 20 and 40% of the total volume of the medium, high values can result in water deficiencies for the plants and lower values can hinder the oxygenation of the plants', impacting directly in the root development (GUERRINI; TRIGUEIRO, 2004; RIGON et al., 2015). Regarding the easily available water (EAW) amount and buffering water (BW), the values taken as a reference in the literature are in the range of 20 to 30% and 4 to 10%, respectively (FERMINO, 2003). In the five treatments evaluated, the results of aeration space (AS) were below the range recommended by the literature (from 20 to 40%), which may cause a lack of oxygen and negatively impact root development (TRAZZI, 2011; ZORZETO et al., 2014). All samples presented values of easily available water (EAW) within the reference range of the literature (from 20 to 30%). Treatments A, B and D showed slightly higher values than treatments C and E. The results of buffering water (BW) were also below the range established as ideal in the literature (from 4 to 10%), however, according to Zorzeto et al. (2014), this characteristic does not directly impact the development of commercial plants, since at the point of BW they would be under water stress.

4.1.2 Seedling development

Table 5 presents the medium values obtained for the plant and roots' heights. The first germination took place six days after sowing (DAS) in the tray with treatment A (100% soil), and the germination of seven seeds was verified. On the seventh day, trays with treatments B, C and D had also germinated seeds. The seeds arranged in trays with treatment E germinated after the tenth day of sowing.

TABLE 5. Plant and roots' height. Source: Authors. * Same letters indicates that there was not a significative difference among the results for p≤0,05

Sample	Plant height (mm)	Root height (mm)
A	19^a ± 1.0	14^a ± 0.9
B	12^b ± 0.7	11^b ± 0.5
C	5^c ± 1.1	12^c ± 0.7
D	9^d ± 0.8	5^d ± 0.7
E	13^b ± 0.7	7^e ± 0.7

Losses were observed in all treatments. Treatments A, B and E had a survival rate of 93.3%. Treatments C and D showed survival rates of 73.3% and 60%, respectively. According to Neto (2011) and Padilha (2016), survival percentages close to or above 80% can be considered high, indicating that the doses of sludge incorporated into the treatments were not harmful to the seedlings. Lobularia marima seedlings reached different heights at the end of the experiment, as shown in Table 5, suffering high influence from the culture medium.

The highest growth in height was observed in treatment A, composed solely of soil, where the seedlings reached, on average, 19 mm. The smallest growth was observed in treatment C, with seedlings with a height average of 5 mm. Figure 6 shows the seedling growth averages for each of the five treatments evaluated. Based on the results, it can be observed that the germination and development of the studied species occurred more quickly in treatment A. This fact may be linked to the physical-chemical factors of the culture medium. As seen throughout the previous section, treatment A had the lowest apparent density and highest moisture content among the five treatments, ideal micropore values, high total porosity, and high-water retention capacity. Furthermore, treatment A also showed the highest conductivity when compared to the other treatments, indicating that there are more soluble salts in this medium. Treatment B, containing 10% sludge, and treatment E, containing 40% sludge also showed satisfactory growth results. It is believed that the development of seedlings in these media may have encountered problems due to the arrangement of the particles, with smaller particles being able to accommodate between the empty spaces, decreasing the porosity and impacting the water retention capacity and aeration space. At the end of the monitoring period, all treatments still had cotyledons, with no evidence of leaves. Treatment A was the one with the best seedling development, followed by treatment B, while treatments C and D showed the worst results, with cotyledons visibly more fragile and yellowish, a fact that may be related to the aluminum content present in the médium (COELHO, 2012; NETO, 2011).

The root development was different for the five studied treatments. Figure 7 shows a comparison between the roots' height. The seedlings of all treatments showed thin and brittle roots. The greatest root length was observed in the treatment A, with an average length of 14 mm. With the increase in the volume of sludge in the treatments, it was noticed a decrease in the size of the seedling roots. The seedlings of treatments B and C showed shorter root length, but close to that of treatment A, with average values of 11 mm and 12 mm, respectively. At last, treatments D and E seedlings presented the shortest roots, with 7 and 5 mm in length, respectively.
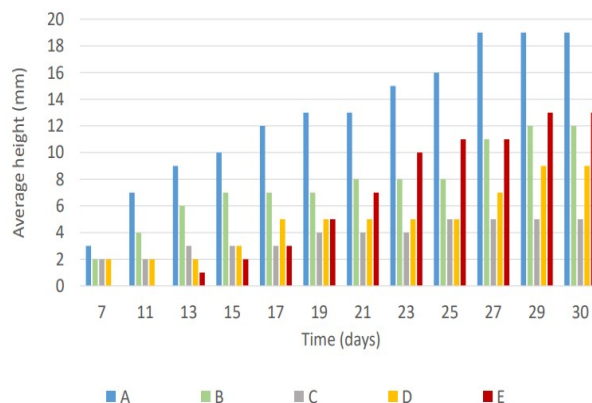

Figure 6 - Average seedling growth for the five treatments evaluated. Source: Authors.

In treatments A, B, and C it was possible to observe small root branches, for treatments D and E these branches were not observed. The low root development can be attributed to the reduced aeration space observed in the treatments, which may have impacted the amount of oxygen available in the culture médium.
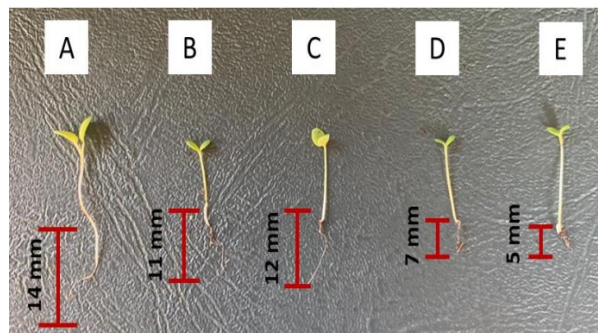

Figure 7 - Root development of species cultivated with the five different types of treatment. Source: Authors

4. CONCLUSIONS

The evaluated residue presents an attractive chemical composition for the application in which it was tested, containing several compounds similar to those present in the soil used and which are beneficial for the land. Calcium and magnesium carbonate, present in a significant amount in the residual sludge, have basic properties, which helps in the correction of the soil pH. This fact could also be observed

through pH analysis, for which treatments with a higher percentage of incorporated sludge showed higher pH. Regarding the salinity of sludge and treatments, it was also possible to perceive a positive relationship, since the salinity present in treatments containing sludge was within the parameters determined by the literature. Through the physical characterization, it was possible to notice that the residue, when incorporated in the soil, caused an increase in density and a reduction in the moisture content of the medium. The granulometric analysis carried out for the sludge and for the treatments demonstrated that the incorporation of the residue causes the reduction of the average particle diameter, which can result in greater compaction and cementation of the material. It was also observed that the increase in residual sludge in the treatments resulted in a reduction in the amount of micropores, which can cause difficulties in aeration, total porosity, which nevertheless remained within the established literary limits and the capacity of water, which, despite being impacted, still showed good results for all treatments. Regarding root and plant development, it was found that the best performance occurred in treatment A, composed entirely of vegetable soil, followed by treatment B, composed of 10% sludge by volume, which also showed satisfactory results. It is believed that the low performance of the treatments is linked to their physical characteristics, since the sludge significantly impacted all physical properties.

Despite the variations found, all treatments showed plantar development, and treatments A, B and E showed high survival rates of the species, above 80%. Based on the data found, it is believed that in lower quantities, up to 10%, the application of the latex paint industry wastewater sludge as substrate for ornamental plants may be a viable alternative for the disposal of the waste in question.

For future studies it is suggested to study treatments with lower quantities of incorporated sludge (between 0 and 10% v:v). It is also suggested to perform ecotoxicity analyzes on the treatments and carry trials with different types of plants, to further evaluate the viability of the reuse of this industrial waste as a substrate.

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129

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130

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131

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133