

**Use of mineral residue from lithium processing as fertilizer in the
cultivation of castor beans (*Ricinus communis* L.)**

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SUMMARY

Research into the characteristics and potential of new sources of fertilizers has expanded, but still deserves study efforts to establish and recommend agronomic methods. This project aimed to use the residue from lithium processing as fertilizer in the production of castor beans (*Ricinus communis* L.). The research included the chemical characterization of residues, development of castor bean plants in terms of assessments of fresh and dry weight of roots, stem, leaves, fruits, plant height, diameter and height of the plant, production of fruits and seeds, determination of the content of oil to diameter and height, and foliar analysis after harvest at 180 days. Experiment in a greenhouse at UFVJM in Diamantina, MG, using a randomized block design (DBC), 2x7 factorial scheme, four replications. 1st treatment consisting of with and without application of 10% of the total applied Aluminum Silicate in Calcinator Furnace Fines (0, 0.1, 0.2, 0.4, 0.6, 0.8 and 1.0t/ ha) + 50% conventional fertilizer. 2nd treatment: seven doses of Aluminum Silicate (0, 1, 2, 4, 6, 8 and 10t/ha). In the soil analysis, there was a greater increase in the levels of K, P and effective CTC of Ca and K using the 1st treatment. In the development and production of castor oil, the 1st treatment with dosages 8 and 10 t/ha + 1.0 t/ha + 50% fertilization showed greater development in phytotechnical analyzes and oil content. The 0 t/ha dosage of the 2nd treatment had the lowest development.

KEYWORDS: Fertilizer; Lithium; Environment; *Ricinus communis* L.; Industrial waste.

1 INTRODUCTION

As the human species advances in the exploration of the environment, to meet population growth and its demands, divergences occur regarding the use of resources and space. Between 2010 and 2022, a record shows that the Brazilian population grew by 6.5% (IBGE, 2023).

The exploitation of natural resources in Brazil began with the arrival of Portuguese colonizers in the 16th century. Brazilwood was one of the first commodities explored, followed by gold and diamond mining in the regions of Minas Gerais and other areas. - 10th century (GOMES, 2021).

Different characteristics were obtained from the intensification of the exploitation of natural resources, both with the industrial revolutions and the increase in technology, together with world trade that changes both the raw material and the most sophisticated products in global demands.

This global issue of natural resources results from the foundation of large-scale production and consumption. The process of exploring the environment is currently responsible for a large part of environmental problems (Brasil, 2001, p. 173).

One of the biggest environmental problems today is found in industrial and mineral activities, mainly in the generation of waste discarded directly into natural systems and with great potential for polluting soil, groundwater and surface waters in the long term. Even with the new technologies developed, in many cases, it becomes practically impossible to eliminate the polluting capacity of some waste even if it is managed, recycled and reused in a way that is harmonious with the environment.

The use of residues in the agricultural environment can be used as an alternative fertilizer in the use of soils poor in nutrients and help to reduce the risk to the environment, with these by-products requiring evaluation of their agronomic performance. Advances in research provide solutions to environmental difficulties related to rock mining, such as generating cleaner alternatives for soil fertilization (DALMORA; et al., 2020).

Brazil's current economy is becoming increasingly costly for rural producers to obtain agricultural inputs. In recent years, the country has experienced a growing increase in fertilizer imports. In 2021, fertilizer imports totaled 39,258,338 tons, volume 12 % higher than the volume recorded in 2020 (ANDA, 2022).

In the coming years, Brazil will account for 50% of food production in the world, with the need and increase in demand for fertilizers being worrying. The country is the fourth largest consumer of fertilizers in the world, corresponding to 8% of this volume. Due to the rise in fertilizer prices, there is a negative impact on exports in the Brazilian trade balance (MAPA, 2022).

In this way, the use of alternative sources of industrial waste and mining processes that have characteristics of corrective, soil conditioner and fertilizers, deposited in yards, landfills and even in the open can be an alternative, not only aiming at the elimination the environmental liabilities of industry and mining, but also the reduction of agricultural production costs that would benefit the entire chain, from producer to consumer.

The accurate study of the physical and chemical parameters of waste from mining companies is essential to guide adequate management of the application of this waste to the soil, aiming at the sustainable use of the area, reducing the risks of contamination of soil and water resources. Characterizing the material allows us to understand its agricultural potential, predicting its effects on the physical, chemical and biological characteristics of the soil as well as plant nutrition. In this study, the mineralogy of the clay fraction and soil texture are fundamental in evaluating nutrient dynamics (Pinheiro, 2002; Oliveira, 2004; Silva, 2004; Matos et al., 1994).

Castor bean (*Ricinus communis* L.) is a plant that belongs to the Euphorbiaceae family, being considered a toxic plant, originating in Ethiopia and spreading throughout the Middle East, India and China. The main castor bean producing countries are India, Mozambique, China, Brazil, Myanmar and Ethiopia, corresponding to 97% of the world's castor bean cultivation. *Ricinus communis* L. has great potential for different areas of use, but the plant's oil accounts for the majority of the use of this crop (ERGUN, 2022).

The properties present in castor oil are seen as an alternative for the production of biodiesel, a biofuel from a renewable and environmentally friendly source (MENDES; et al., 2020).

Due to the potential use of castor oil in the form of biofuel, cultivation has increased, but production is still low when compared to other oilseeds. Therefore, further research is necessary with different forms of production linked to the use of by-products arising from mineral exploration, as a way of reusing and using renewable sources for the benefit of the environment.

2 OBJECTIVES

This work aimed to use waste from mineral exploration and lithium processing as a conditioner-fertilizer in the cultivation of castor beans (*Ricinus communis* L.) and as a way of using mining by-products in agriculture.

3 METHODOLOGY

3.1 Characterization of the experimental area

The experiment was conducted during the months of April to October 2022, in a greenhouse at the Department of Agronomy – DAG/UFVJM, in Diamantina – MG, on the Campus of the Federal University of Vales do Jequitinhonha e Mucuri - UFVJM, located in the municipality of Diamantina, Minas Gerais, Rodovia MGT 367 – Km 583, nº 5000 – Alto da Jacuba.

Under geographic coordinates longitude 43° 36' W, latitude 18° 15' S, 1296 m altitude. The local climate is characterized as tropical, with a dry winter season (Aw), and a dry period from April to September (KÖPPEN and GEIGER, 1928).

3.2 Acquisition of materials

The experiment was carried out using 2 types of waste generated from the processing of Lithium from Companhia Brasileira de Lítio located in the city of Divisa Alegre in Minas Gerais, under geographic coordinates longitude 41 ° 20' W, latitude 15° 43' S. The waste was collected in the company's disposal areas, bagged and transported to the UFVJM Campus, located in Diamantina-MG, to carry out soil experiments and plant development. In the laboratory, the materials were dried in the shade, crumbled and sieved through 0.2 mm mesh sieves to homogenize their particles.

These two materials were called Waste 1: Aluminum Silicate and Waste 2: Fines from the Calciner Furnace.

3.3 Soil and waste preparation

The experiment was carried out in a randomized block design (DBC), in a 2x7 factorial scheme, with four replications. There are 2 types of fertilizers and 7 doses: the first fertilizer was composed of seven different types of doses of Aluminum Silicate (0, 1, 2, 4, 6, 8 and 10t/ha) and the second fertilizer was composed of with and without the application of 10% of the total applied Aluminum Silicate in Calcinator Furnace Fines (0, 0.1, 0.2, 0.4, 0.6, 0.8 and 1.0t/ha) associated with half of the conventional fertilizer recommendation. This application and distribution of treatments in t/ha were carried out empirically since there is no record of the use of lithium waste as fertilizers in the literature.

To calculate conventional fertilizer recommendations for castor bean cultivation, it was carried out in accordance with the recommendations of the Soil Fertility Commission of the State of Minas Gerais - CFSEMG (1999). 0.183g of Triple Superphosphate and 0.127g of Potassium Chloride were applied. For top dressing, 0.500 g of Ammonium Sulfate were used.

The calculation for the application of waste was carried out using the proportionality of the soil volume of one hectare at a depth of 10 centimeters, corresponding to 1,000 m³ of soil in relation to the volume of soil in the pot (5.0 liters). The same was done for the application of conventional fertilizers.

Pots with a capacity of 5 liters were used, and the blocks were composed of 28 pots each, with 2 castor bean plants, totaling 112 pots.

The data obtained from the phytotechnical analyzes were analyzed by regression, and the chemical analyzes and elemental analysis were analyzed using the Principal Component Analysis (PCA) technique and the statistical program SISVAR version 5.6, which were subjected to analysis of variance by T test and comparison of means using the Tukey test at 5% significance.

3.4 Experiment setup

After weighing the materials (Aluminum Silicate Residue and Fine Residue from the Calcinator Furnace), and filling the pots with soil, they were applied individually to the soil in the pots and mixed in plastic bags to make the materials uniform with the soil. The soils were then returned to the pots and irrigated to begin the reaction process. In this experiment, castor bean (*Ricinus communis* L.) was planted, a plant from the Euphorbiaceae family that produces oil.

Four seeds were sown per pot and after the start of germination, thinning was carried out, keeping two plants per pot. Nitrogen top dressing was applied to all pots, twenty days after seed germination. During the process of conducting the experiment, the pots were initially irrigated every two days with the aid of a 500ml Becker, irrigation was done by applying 200ml of water per pot, during growth the waterings became spaced 3 by 3. days so that water stress does not occur.

3.5 Analysis using the Energy Dispersive X-ray Fluorescence (EDXRF) technique

The EDXRF technique was carried out at the Multiuser Laboratory – LIPEMVALE, at the Federal University of Vales do Jequitinhonha and Mucuri - UFVJM, Diamantina – MG with the purpose of obtaining the chemical elements present in the waste generated from the extraction of lithium and used as fertilizers in this work. The equipment used in this procedure was the X-ray Fluorescence Spectrometer, model EDX 720 (Shimadzu).

In the analysis carried out in this work, only atmospheric air was used, restricting the detection of metals between Al ¹³ and U ⁹² . The software used was PCEDX, version 1.11 Shimadzu. EDXRF analyzes were carried out with samples of the two residues used (Aluminum Silicate and Calcinator Furnace Fines) in which the materials were placed in sample holders made with polypropylene film and the analysis conditions were as follows: 10 mm collimator, scans with voltages of 0-40 KeV (Ti-U) and 0-20 KeV (Na-Sc) with a time of 100 sec. each

The analyzed material was hit with an X-ray beam that interacted with the atoms in the sample, causing the ionization of the innermost layers of the atoms. The filling of the resulting vacancies, by more peripheral electrons, induced the emission of X-rays characteristic of the constituent elements of the sample.

3.6 Phytotechnical analyzes

The evaluations were carried out 180 days after planting the seeds. *Ricinus communis* L. plants were collected and separated into root, stem, leaf and fruit. The agronomic characterization of height was initiated after planting and carried out throughout the

development of the plants. Plant height was determined using a 60 cm ruler and their diameter was measured with a digital caliper at the initial portion of the stem.

After weighing and measuring the green weight, they were packaged in kraft paper bags, identified and then dehydrated in an oven at 65° C until reaching a constant weight. Using a precision digital scale, the weight of total fresh mass (TFM) and total dry mass (TDM) was determined in g.plant⁻¹. Seed samples were collected to determine oil content and total plant production.

3.7 Soil and leaf chemical analyzes

3.7.1 Soil analysis

After dismantling the experiment, soil samples were collected from each pot to carry out chemical analyses. The samples were taken to the Multiuser Laboratory – LIPEMVALE, at the Federal University of Vales do Jequitinhonha and Mucuri - UFVJM, Diamantina – MG, where they were dried and sieved. Soil chemical analyzes were determined using standard methods (EMBRAPA 1997). To extract phosphorus, potassium and micronutrients, an extractor with Mehlich-1 solution was used, while exchangeable calcium, magnesium and aluminum were extracted with a 1 mol L⁻¹ KCl solution.

Phosphorus was determined colorimetrically using spectrophotometry, and all other nutrients were determined using atomic absorption spectroscopy (MORAES and RABELO, 1986). To determine exchangeable Al, the volumetric method was used by titration with sodium hydroxide, after extraction of Al³⁺ from the soil by 1 mol L⁻¹ KCl.

To determine pH measured in water, 10 cm³ of soil was mixed with 50 ml of water, shaken and pH measured with an electrode touching the bottom of the bottle. The SMP pH was determined by adding the buffer solution after determining the soil pH, with the reading being taken in the supernatant, so that the electrode bulb slightly touches the sedimented soil at the bottom of the bottle.

3.7.2 Leaf chemical analysis

The plant material (roots, stem and leaves) was identified and taken to the Multiuser Laboratory – LIPEMVALE, at the Federal University of Vales do Jequitinhonha and Mucuri - UFVJM, Diamantina – MG, where it was dried in an oven at 65°C until it acquired a constant weight. After drying the material, it was crushed in a mill, and homogeneous samples weighing 0.500 g were taken. These samples were digested with nitric acid (HNO₃) in a closed system using PFA bottles with controlled temperature and microwave radiation power. Phosphorus was determined colorimetrically using spectrophotometry and the other nutrients were determined using atomic absorption spectroscopy (MORAES and RABELO 1986).

3.8 Analysis using the Elementary Analyzer technique

The LECO® CHNS/O Elemental Analyzer technique, model TruSpec Micro, was carried out at the Multiuser Laboratory – LIPEMVALE, at the Federal University of Vales do Jequitinhonha and Mucuri - UFVJM, Diamantina – MG with the purpose of quantifying the elements present in castor bean plants. To obtain these analyses, plant parts (roots, stems and leaves) were crushed in a mill and sieved through a 0.2 mm fine mesh sieve and weighed into micro-capsules. Then the capsules were attached to the equipment carousel where they are incinerated at 1075 °C in a quartz tube for the elements Nitrogen, Protein, Carbon, Hydrogen, where the gases generated are quantified in an infrared detector.

3.9 Seed oil content

The determination of the oil content in castor seeds was carried out in the Biomass Cerrado Technology laboratory – LTBC at the Federal University of Vales do Jequitinhonha and Mucuri - UFVJM, Diamantina – MG, through chemical extraction using organic solvent, using the Soxhlet method with Ethyl Ether, based on the procedures defined by the Association of official Analytical Chemists (AOAC, 1995). The seed samples were ground in a mortar and placed in porous cartridges. The cartridges were placed in the extraction chamber that is suspended above the volumetric flask containing the solvent, and below a condenser. The flask is heated and evaporates the solvent that moves in the gaseous phase towards the condenser, which is converted into a liquid that drips into the cartridge containing the sample. The extraction lasted an average of four hours.

The oil content was determined by the percentage gravimetric relationship between the oil obtained and the seeds subjected to extraction (BALIZA *et al.*, 2004).

4 RESULTS AND DISCUSSION

4.1 Waste Characterization

By carrying out the analyzes using the X-Ray Spectrophotometer, it was possible to identify the presence of minerals in both wastes (Table 1), and also the highest percentages of minerals present in the material (Table 1). In the residue called Aluminum Silicate, Si (47.65%), Al (33.10%), K (7.28%) and Fe (5.83%) were found in higher proportions. While in the residue called Calcinator Furnace Fines, Si (43.44%), Al (31.27%), Ca (14.95%) and S (7.64%) were found.

The presence of the macronutrients P, K, Ca, S and the micronutrients Fe, Mn, Zn (Table 1) is necessary to meet the nutritional demands of agricultural crops and can influence all stages of the crop cycle. A study carried out by (Dalmora *et al.*, 2020) in Triunfo - RS, Brazil, showed the analysis of the by-product of volcanic rock (andesitic vesicular rock) and field experiment as potential fertilizer. The presence of Si, Al, Ca, P, K, among others identified as a possible resource of micro and macronutrients for agricultural production and reduction of environmental impacts, was evaluated.

Table 1 - Main minerals found in the X-ray fluorescence spectrometer analysis

Aluminum Silicate		Calcinator Furnace Fines	
Minerals	%	Minerals	%
Si	47.65	Si	43.44
Al	33.10	Al	31.27
K	7.28	Ca	14.95
Fe	5.83	S	7.64
Ca	2.93	Fe	1.90
S	1.01	K	0.45
P	0.90	Mn	0.24
Ti	0.47	Sr	0.06
Mn	0.36	Rb	0.03
Rb	0.20	Ga	0.02

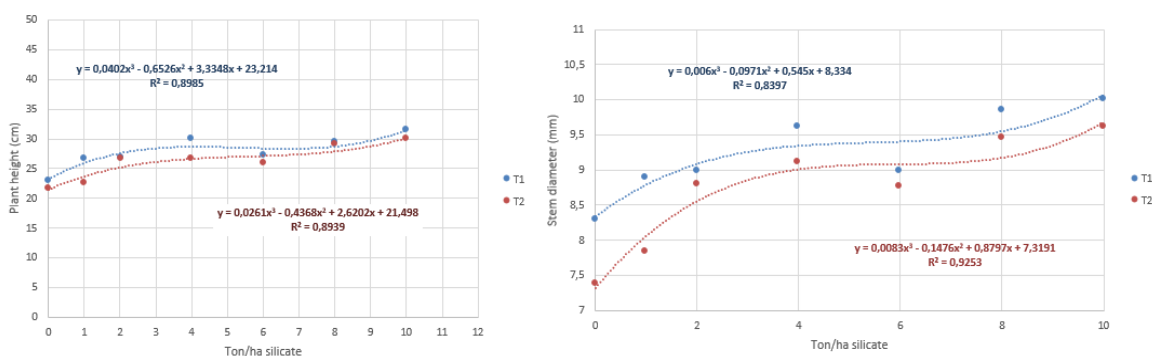
Source: Authors, 2022.

4.2 Phytotechnical analyzes

Significant differences were observed in the parameters of plant height and diameter (Figure 1), demonstrating that the greater the dosage amount of silicate residue and fines from the calciner furnace, the greater the development of plants in both treatments.

The 1st treatment using doses of aluminum silicate residue + fines from the calciner oven + 50% of conventional fertilization obtained higher averages than the cultivation of the 2nd treatment which used only aluminum silicate residue. The increase in these parameters in the treatments may primarily be related to the increase in dosages, since the plants achieved linear growth.

Figure 1 – Plant Height Chart; Stem diameter (Two plants – 180 days)



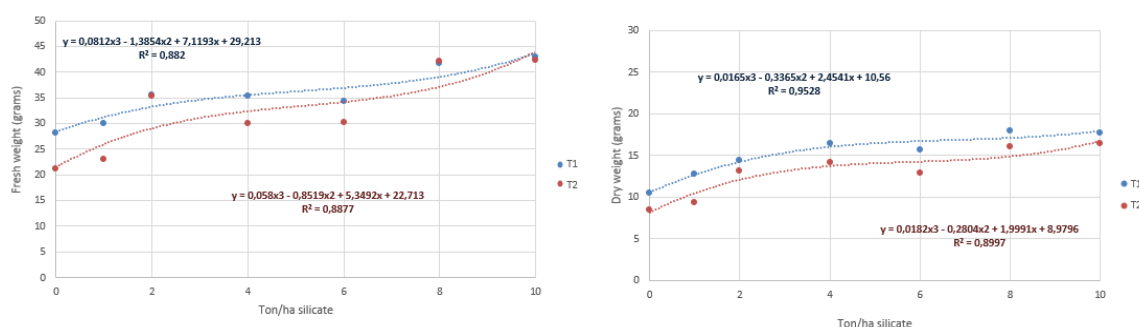
Source: Authors, 2022 .

Due to the increasing dosages of Waste applied to the soil, both in TFM and TDM (Figure 2), it is possible to observe, according to the coefficient of the adjusted equations, that the plants showed a similar tendency to behave.

Regarding the greater quantity of Fresh and Dry Mass, it is possible to observe that Treatments of 8 and 10 tons of Silicate Waste demonstrated greater growth. There was a higher

yield of fresh and dry matter in the treatments using 8 and 10 ton /ha of silicate residue + furnace fines + 50% of conventional fertilization (Figure 2). This result may probably have occurred due to adverse interactions between excess P and K and other nutrients in the waste used, which is confirmed by the analysis of the increase in nutrients in the table in Annex A (CARVALHO *et al.*, 2018).

Figure 2 – Total Fresh Mass Chart (TFM); Total Dry Mass (TDM) – Two plants (fruit, stem, root and leaf)



Source: Authors, 2022.

Results found by Ramos *et al.*, (2019), in which they carried out a study on the potential fertilizer containing Ca, Mg, K and P in dacite rock residues, and testing the cultivation of corn and black oats for 70 days each, with different doses of residue showed that the K available from the residue was available to the plants.

According to SOARES, in 2006 there are few times when an increase in productivity is observed as a result of the application of potassium in research carried out both in Brazil and in other countries. The castor bean plant possibly needs low levels of this element for the normal functioning of its physiology and has a great capacity to absorb this nutrient from the soil, according to soil analysis.

4.3 Soil and leaf chemical analyzes

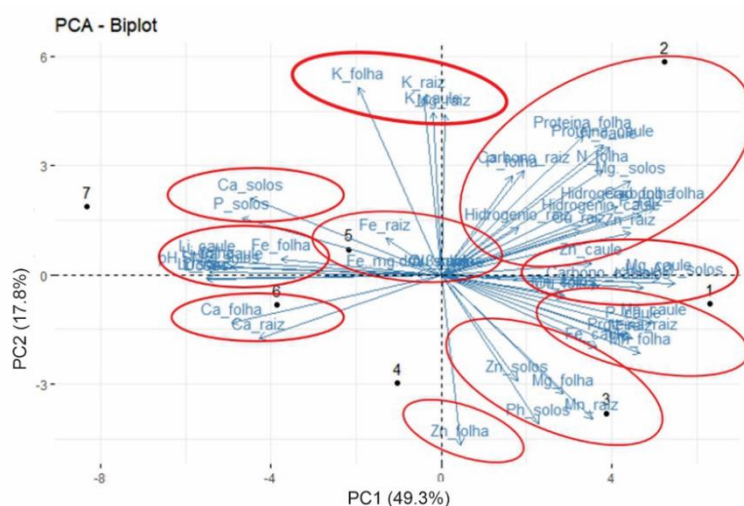
To evaluate the effect of treatments on chemical attributes, multivariate Principal Component Analysis (PCA) was used, considering standardized data.

PC1 (figure 3) contributed up to 49.3% of the total variance and PC2 contributed up to 17.9% of the total variance. Differences between treatments were mainly controlled by PC1. Treatments with dosages of 1 and 2 t/ha of Silicate Waste associated with 0.1 and 02 t/ha of kiln fines with 50% conventional fertilization had better interference and changes in the analyzed components.

In the first treatment, although the PCA results demonstrated greater interference from fertilizers in dosages 2 (1t/ha) and 3 (2t/ha) (figure 3), it does not mean that they were the best dosages in the 1st treatment, since these two dosages did not obtained greater growth (figure 2) and oil content (figure 5). Dosage 7 (10 t/ha) showed that there was interference with Ca and P, demonstrating that the increase in these nutrients helped to increase productivity (figure 5)

and consequently loss of reserves of other nutrients, resulting in lower reserves in the soil. and in the aerial part.

Figure 3 – Score map of principal component analysis (PCA) of chemical elements under different combined applications of silicate residue + furnace fines + 50% conventional fertilizer

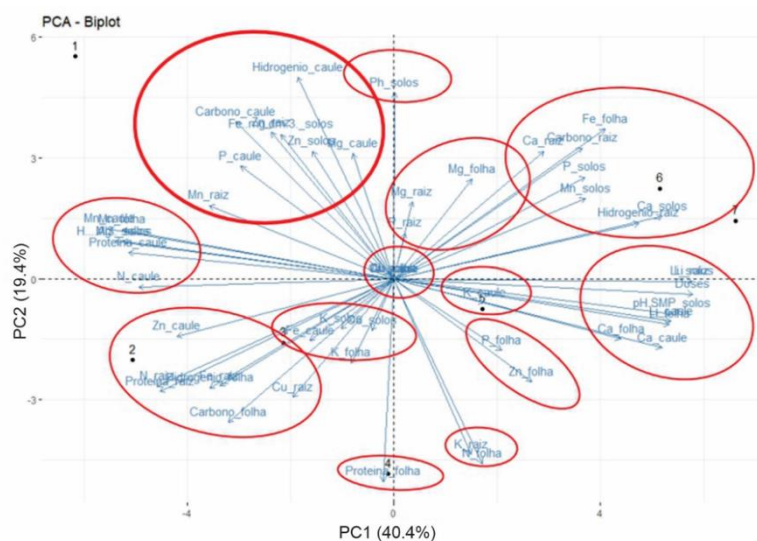


Source: Authors, 2022.

The results of the research demonstrated that the element K and Ca underwent changes, but not being a specific treatment, this may need to be adjusted and optimized according to the dosage used to achieve high quality and high yields.

Nutrient K was found in both treatments. Potassium (K) is a necessary and important mineral, as it contributes to increasing the productivity of several crops (CARVALHO et al. , 2018). The presence of macronutrients (P, K, Ca, S) and micronutrients (Fe, Mn, Zn) demonstrates that the minerals present in the residues (Table 1) became available to castor beans, influencing their development.

Figure 4 - Principal component analysis (PCA) score map of chemical elements under different Silicate Waste applications.



Source: Authors, 2022.

PC1 (Figure 4) contributed up to 40.4% of the total variance and PC2 contributed up to 19.4% of the total variance. Differences between treatments were mainly controlled by PC1. The PCA results showed that in the 2nd treatment, dosages 1 (0 t/ha) and 2 (1 t/ha) stood out the most in relation to the other dosages (Figure 4).

Dosages 6 and 7 did not have greater interference between them and there was also no greater fruit production (Figure 5). Demonstrating that the use of silicate waste alone does not increase productivity.

Dosage 1 (0 t/ha) had the elements that had the greatest interference: Carbon (stem), Hydrogen (stem), Zn (root), Zn (soil) Mg (stem), Mn (root), P (stem). The research result demonstrated that elements such as Zn (stem), Cu (root), Carbon (leaf), Protein (root), Fe (root), N (root), underwent changes in dosage 2, which demonstrates the need adjustment for its use as a possible fertilizer.

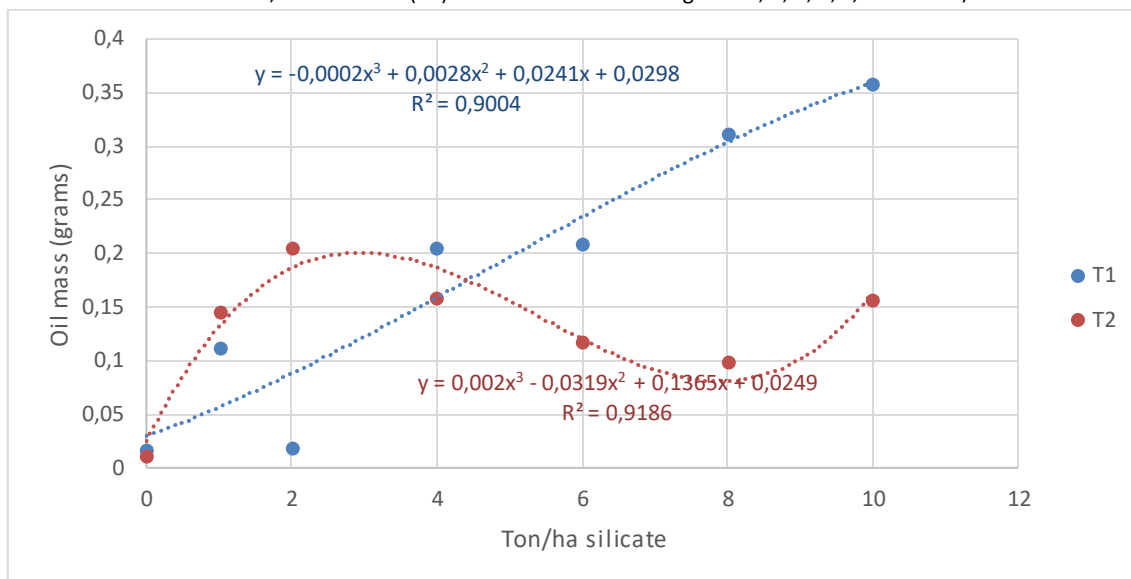
Based on the data presented in this work, we can conclude that the use of these residues as fertilizer can bring significant benefits to castor bean production. Furthermore, the use of residue as fertilizer can contribute to reducing the environmental impact of castor bean production (FERREIRA *et al.*, 2013).

4.4 Oil content

The oil content of castor seeds varied between the treatments studied, according to data presented in the summary of the graph analysis (Figure 5). Moreira *et al.*, (2008), found high variability in weight and oil content in castor bean cultivars, corroborating the results found in this study.

The average seed oil content per treatment demonstrated that the highest castor oil content occurred in treatments 6, 8 and 10 ton/ha of aluminum silicate with fines from the calciner oven and with half the conventional fertilization (Figure 5).

Figure 5 - Oil content of castor seeds (average of two plants) collected 180 days after sowing in soils in comparison of treatments: Treatment 1 (T1): Silicate residue + fines from the calciner oven + 50% conventional fertilization at dosages of 0, 1, 2, 4, 6, 8 and 10 t/ha of Silicate Waste + 0.1, 0.2, 0.4, 0.6, 0.8 and 0.1 t/ha oven fines + 50% conventional; Treatment 2 (T2): Silicate residue at dosages of 0, 1, 2, 4, 6, 8 and 10 t/ha.



Source: Authors, 2022.

According to Oliveira (2004), it is necessary for there to be availability of macro levels of N, P, K, Ca, Mg and S and micronutrients B, Cu, Fe, Mn, Mo and Zn suitable for the castor bean flowering season, such a fact justifies the higher oil contents due to treatments at the highest dosages of silicate residues and calciner furnace fines.

According to Beltrão (2003) the average crude oil content for castor beans varies from 35% to 55%. Thus, the average oil content of the castor bean plants that produced seeds from the 1st treatment at dosages 8 and 10 (Figure 5) are similar to those found in the literature, demonstrating the feasibility of using these residues as fertilizers.

5 CONCLUSIONS

It is concluded from this work that *Ricinus communis* L. presented different responses to the treatments to which it was subjected. Cultivation with the highest dosages of the 1st treatment (silicate residue + autofurnace fines residue + 50% of the recommended conventional fertilizer dose) obtained better results. The results found in this work show that castor bean plants, when cultivated with dosages of 8 and 10 ton /ha of residual silicate, with the application of autofurnace fines associated with partial chemical fertilization (50% of the recommended dose), had a positive influence on both growing in terms of oil production. The results of the phytotechnical analysis indicated that the use of the conditioner-fertilizer promoted a significant increase in the fresh and dry weight of castor beans, as well as in the height of the plants and the diameter of the stem at the base of the stalk.

In the development and production of castor oil, the 1st treatment with dosages 8 and 10 t/ha + 1.0 t/ha + 50% fertilization showed greater development in phytotechnical analyzes and oil content. The 0 t/ha dosage of the 2nd treatment had the lowest development. autofurnace fines residue + 50% of the recommended dose of conventional fertilizer) are similar to those found in the literature, demonstrating the viability of use of these residues as fertilizers. Finally, the use of waste from mineral exploration and lithium processing as a conditioner-fertilizer in castor bean cultivation presents a promising potential to increase the yield and quality of plants, in addition to contributing to the reduction of environmental impacts caused by inadequate disposal of this product. residue. However, more studies are needed to evaluate the economic viability of using this conditioner-fertilizer on a large scale.

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