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Original Article

Variants in the neutralization of fish oil to obtain effluent with fertilizing properties

Variantes na neutralização de óleo de peixe para a produção de efluentes com propriedades fertilizantes

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Abstract

Currently, an important trend in the agricultural activity is the development of organic agriculture. In this context, the application of a fertilizer derived from an effluent of the oilseed industry, to beet (*Beta vulgaris* L.), is evaluated, with the objective of improving the agronomic characteristics of beet and complementing the chemical deficiency of the soil in OM and N. The modified effluent is obtained from fish oil wash water, which contains nutrients, highlighting K, P and N. The experiment was conducted in a crop field, using completely randomized block design, with five treatments: T1, T2, T3, T4 and T5 with fertilizer doses of 0, 2, 3, 3, 4, 5 L/ha, respectively, applied to the beterraga. The results show a modified effluent with fertilizer properties, particularly for its high concentration of N (25.76 g/L) and its content in K and P. By changing the neutralizing agent and other complementary chemicals intended to confer fertilizer properties to the effluent produced in the oil refining process, a fertilizer was obtained that was applied to the beet crop in five treatments, highlighting the fourth treatment T4 with a yield of 23.97 t/ha.

Keywords: *Beta vulgaris* L., neutralization, modified effluent.

Resumo

Atualmente, uma tendência importante na atividade agrícola é o desenvolvimento da agricultura biológica. Neste contexto, é avaliada a aplicação de um fertilizante derivado de um efluente da indústria de oleaginosas, à beterraba (*Beta vulgaris* L.), com o objetivo de melhorar as características agronômicas da beterraba e complementar a deficiência química do solo em MO e N. O efluente modificado é obtido a partir da água de lavagem do óleo de peixe, que contém nutrientes, destacando-se K, P e N. A experiência foi conduzida num campo de cultivo, utilizando o desenho de blocos completamente aleatórios, com cinco tratamentos: T1, T2, T3, T4 e T5 com doses de fertilizantes de 0, 2, 3, 4 e 5 L/ha, respectivamente, aplicados na beterraba. Os resultados mostram um efluente modificado com propriedades fertilizantes, nomeadamente pela sua elevada concentração de N (25,76 g/L) e pelo seu teor em K e P. Alterando o agente neutralizante e outros químicos complementares destinados a conferir propriedades fertilizantes ao efluente produzido no processo de refinação do petróleo, obteve-se um fertilizante que foi aplicado à cultura da beterraba em cinco tratamentos, destacando-se o quarto tratamento (T4) com uma produtividade de 23,97 t/ha.

Palavras-chave: *Beta vulgaris* L., neutralização, efluente modificado.

1. Introduction

The current paradigms in the field of organic agriculture and environmental protection are constantly evolving, raising new theories and technological applications that seek to improve the quality of life. Anthropogenic impacts increasingly drive ecological and evolutionary processes at many spatio-temporal scales, demanding greater capacity to predict and manage their consequences (Thrall et al., 2011). In the coming few years, one of the major challenges for the vegetable industry will be to ensure sufficient production for the increasing world population in a sustainable way, with improved water and nutrient use efficiency (De Pascale et al., 2018). With its high nutrient content, waste and by-products originating from fish and seafood industry (including aquaculture) are one of the most promising candidates to produce alternative fertilising products which can play a crucial role to replace synthetic mineral fertilisers (Zhang et al., 2023).

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aquaculture) are one of the most promising candidates to produce alternative fertilising products which can play a crucial role to replace synthetic mineral fertilisers (Hammoutou et al., 2017). The use of effluent from the edible oil industry was investigated, in this case the effluent resulting from the wash water in the fish oil refining process, which is discharged into the sewerage system. A limiting factor for the reuse and recycling of treated wastewater from the fish canning industry in an industrial plant and also for other uses is the high salt content, which persists even after conventional treatment (Cristóvao et al., 2015)

The activity of the oil industry has a negative environmental impact since, in the fish oil refining stage, effluents are generated that pollute water resources (Cherrepano et al., 2022). Given this reality, it is necessary to develop research that scientifically evidences solutions to this problem, in this purpose it is necessary to seek strategies to improve the sustainability of their wastewater treatment processes (Almeida et al., 2022).

In the case of manufacturing activities that process food, wastewater with a high organic load requires treatment. In this regard, companies could make variations in their industrial processes to contribute to the development of viable processes, not only economically but also environmentally. In relation to these approaches, it is indicated that wastewater from palm oil mills or Palm Oil Mill Effluent (POME) requires treatment prior to its use in irrigation or its discharge into surface waters (Althausen, 2016). Wash water from fish oil refining contains macronutrients such as N, P, K and micronutrients that can be used as a foliar fertilizer solution for vegetables according to laboratory analysis results from the National Institute for Agrarian Innovation-Huaral (NIAI-H). The biofertilizer increased the microbial fertility of the soil expressed as the number of colony-forming units of nitrogen-fixing and phosphate-solubilizing microorganisms per gram of soil (Maquén et al., 2023). Foliar fertilization makes it possible to quickly provide plants with essential nutrients, mainly micronutrients, which can significantly improve the quality of yields (Zydlik et al., 2022).

The advantage of using KOH in petroleum refining is to produce a biofertilizer with nutrient properties instead of a polluting effluent. In organic farming, biofertilizer usage is prioritized over chemical fertilizers utilized in conventional farming (Rani et al., 2023).

The objective of the work was to evaluate the feasibility of making a change in the neutralizing agent in the refining of edible oil, to obtain a liquid fertilizer instead of obtaining a harmful effluent to the water resource, the fertilizing properties of the product obtained are evaluated by applying this fertilizer to the beet crop in a randomized block design system in a crop field, another objective of this research was to reduce soil deficiency in OM (organic matter) and soil N, with the application of the fertilizer obtained.

2. Materials and Methods

2.1. Chemical reactions to obtain fertilizer

The effluent was prepared at laboratory level from a sample of crude fish oil, which was previously degummed with phosphoric acid, which had a free acidity of 2%, so it was treated with KOH, changing the neutralization normally performed with NaOH. The result of the chemical reaction is shown in Equation 1.

$$\begin{aligned} &COOH-\left(CH_2\right)_7-CH=CH-\left(CH_2\right)_7-\\ &CH_3+\ KOH\ \rightarrow\ COOK-\left(CH_2\right)_7-\\ &CH=CH-\left(CH_2\right)_7-CH_3+\ H_2O \end{aligned} \tag{1}$$

The neutralized oil was separated using a decanting bulb, to separate the salts formed from the free fatty acids present in the oil mass and the neutralizing agent, obtaining soap and alkaline water. The soap formed is split by adding H_2SO_4 at 98% concentration, producing an acidic aqueous mixture, according to the following chemical reaction, which is shown in Equation 2.

$$\begin{aligned} &COOK-\left(CH_2\right)_7-CH=CH-\left(CH_2\right)_7-\\ &CH_3+\ H_2SO_4\rightarrow\ COOH-\left(CH_2\right)_7-\\ &CH=CH-\left(CH_2\right)_7-CH_3+\ K_2SO_4 \end{aligned} \tag{2}$$

The resulting alkaline wash water was mixed with the acidic water produced in the soap splitting process. This mixture was treated with ammonium hydroxide to add N to the mixture and at the same time neutralize the mixture obtained, according to the following chemical reaction, which is shown in Equation 3.

$$H_2SO_4+\ NH_4OH\ \rightarrow\ SO_4\left(NH_4\right)_2+\ H_2O \tag{3}$$

In the chemical neutralization reaction shown, the aqueous mixture, acidic due to the H_2SO_4 present in the effluent, was neutralized with NH_4OH to a neutral pH, generating a fertilizing aqueous solution, which was given the name of modified effluent (ME).

2.2. Factor of study

The application of the ME doses, which was obtained at laboratory level, of fertilizer properties, by the addition of chemical inputs in its chemical processing, such as KOH, H_2SO_4 and NH_4OH, producing 5 L of this ME, which was applied in the experimentation of the beterraga crop, taking into consideration the chemical analysis of soil and ME, the dosages were estimated for the five treatments, where the T1 treatment corresponds to the control test, and for the T2, T3, T4 and T5 treatments, doses were used: 2, 3, 4 and 5 L of this ME per 200 L of water respectively. Biofertilizer application from fish waste at 1, 1.25 and 1.5% increased height, root length and yield of cowpea (Maquén et al., 2023).

Prior to beet planting, the soil was prepared in a conventional manner, i.e., weeding, construction of contour furrows, moistening of the soil and finally sowing of the seeds of the beet variety "Early wonder tall top". Then the experimental area was delimited in three blocks, where five treatments were carried out for each of the blocks. Planting was done uniformly in all plots with a distance of 0.10 m between plants and 0.60 m between furrows, where after 10 and 15 days the proposed treatments were applied. After the doses were applied, the agronomic characteristics of the beet crop were evaluated until harvest, and the data recorded were evaluated by means of the analysis of variance and Duncan's test.

For foliar analyses for each treatment, leaf samples (100 g of leaves for each treatment) were taken at harvest from the central part of the plant and taken to the laboratory of the NIAI-H.

2.3. Experiment

The experiment was carried out in the Medio Mundo population center located in the district of Vegueta, province of Huaura in Peru. The crop field is located on the coast of the Pacific Ocean with an altitude of 19 m and with geographical coordinates: Latitude: -11.0233, Longitude: -77.6442, 11° 1′ 24″ South, 77°38′39″ West. It presented an average temperature ranging between 19-22 °C and a relative humidity between 77-85%. In the research, samples from each plot were evaluated and processed by statistical analysis, which allowed determining which treatment stood out in relation to the others. The population refers to the beterraga plants that grew from 0 to 19 meters above sea level in the selected field. The sample of 16 plants was taken from a total of 64 plants per treatment, from the central furrows in order to avoid the edge effect. For data collection, observation and quantification techniques were used to evaluate the physical characteristics of the beet crop.

2.4. Statistical analysis

The data obtained from the three blocks were processed by analysis of variance (ANOVA) adopting a confidence level of 95%. The analysis of variance was processed with the statistical package InfoStat (version 2017) applying the Duncan comparator at 5% error, which determined the statistical significance in the averages of the treatments. Likewise, it was determined which treatment stood out with respect to the others.

3. Results and Discussion

3.1. Improvement of soil deficient in OM and N

According to the soil analysis carried out at NIAI-H, shown in Table 1, it was determined that there was a slightly alkaline pH, low concentrations in OM and N, high in P and adequate in K, according to the established values (Prialé, 2016). The optimal pH range for nutrient availability, microbial activity, and soil structure is between 6.0 and 7.0 (Dewangan et al., 2023). Previous research has demonstrated that the cation exchange capacity (CEC) of soil and the balance of exchangeable cations Ca, Mg, and K are key factors affecting plant growth and development. We hypothesized that balancing exchangeable cations based on increased CEC would improve plant growth and development (Yang et al., 2024).

Therefore, this soil is suitable for planting vegetables; however, it is necessary to apply OM, N and optimal irrigation drainage. Among the biological activities of the soil, the most important are related to OM and nutrient cycling, which allow us to detect, together with other physical parameters of the soil, early changes in its biochemistry, degree of soil compaction and biological activities produced by anthropogenic impact (Alvear et al., 2007). On the other hand, if the N content is low, the rate of OM decomposition decreases, and the rate of C mineralization will depend on the addition of nitrogen sources (Ferrera and Alarcón, 2011). On the other hand (Torres et al., 2017) concluded that production can be increased by improving the efficiency of fertilization that includes OM. The soils we used were relatively nutrient-rich prior to the application of the fish effluent, which may be responsible for the limited impact, and differences may be more apparent in acidic and nutrient-poor soils or when fertigation is used over a longer duration (Cerozi et al., 2022).

3.2. Modified effluent analysis

According to the results shown in Table 2, it was determined that there was a higher concentration of total residues, high in N, but low in P, K, Ca, Mg and C/N ratio. N is the most limiting nutrient for the production

Table 1. Soil chemical analysis of the experimental area.

EC 1:2:5 mS cm^{-1}	pH 1:2:5	OM %	N %	P ppm	K ppm	$CaCO_{3\%}$	Exchangeable cations (mEq/100 g soil)				CEC
							Ca	Mg	Na	K	
0.73	8.0	0.51	0.03	17.05	115	5.72	5.95	1.76	0.09	0.28	8.08

CEC = Cation exchange capacity; OM = organic matter; EC = electrical conductivity.
Source: INIA (2021a).

Table 2. Complete chemical analysis of modified effluent.

ID Sample	pH	EC mScm^{-1}	Total solids g/L	OM	N	P	K mgL^{1}	Ca	Mg	C/N
Liquid fertilizer	8.24	471	359	722	25760	88.86	1707.5	17.19	1.85	0.03

Source: INIA (2021b).

of vegetable crops, but anthropogenic sources pose risks due to its transformation into several reactive forms and movement throughout the environment (Valenzuela, 2024). The pH of the effluent is alkaline and its EC is high, which indicates that it is a moderately saline effluent, so it is analyzed that the effluent has a concentration of salts, adequate in N and low in other nutrients. In conclusion, the wastewater from fish farms is good sources of fertiliser to the farmlands (Musa et al., 2020)

Significant changes in soil chemical attributes occurred due to wastewater application. Irrigation with wastewater did not change the pH or the concentration of Fe and Zn in the soil (Barreto et al., 2013). Secondary and tertiary treated agro-industrial wastewaters were characterized by higher plant nutrient contents, such as NH_4 -N, NO_2-N, PO_4^{2-}, K^+, Ca^{2+}, Mg^{2+}, TSS and organic matter (Tarantino et al., 2017).

However, the low nutrient concentrations of the soil used, as shown in Table 1, were compensated by the nutrient contribution of the amended effluent, which was observed in the results in the yield of the beet product. In the fish farm treatment, shoot fresh weight increased by 203% and 250% compared to river water irrigation in basil and purslane, respectively. N, P, K, Cu and Mn concentrations in basil were significantly increased in the farmed treatment compared to river water irrigation. Therefore, the use of fish farm effluent in irrigation satisfies the water requirements of plants and can also improve the availability of nutrient elements for basil and purslane (Kaab et al., 2019)

The irrigation water used for the experimental crops shown in Table 3 has medium salinity, suitable for irrigation. In certain cases it may be necessary to use excess volumes of water and to use salinity tolerant crops. Water with low Na concentration is suitable for irrigation in most cases.

Table 3. Complete chemical analysis of the water used in the experiment.

Determinations		
EC ms cm^{-1} a 25 °C		601.00
pH		8.26
Ca^{2+}	meq/L	3.36
Mg^{2++}	meq/L	1.21
Na^+	meq/L	1.23
K^+	meq/L	0.25
Sum of Cations		6.05
CO_3^{2-}	meq/L	0.00
HCO_3^-	meq/L	2.50
Cl^-	meq/L	2.10
NO_3^-	meq/L	1.50
SO_4^{2-}	meq/L	0.12
Sum of Anions		6.22
Fe	ppm	0.014
Zn	ppm	ND
Cu	ppm	0.036
Mn	ppm	0.092
SAR		0.81
Classification		C2-S1

Source: INIA (2021c).

3.3. Evaluation of plant agronomic variables

As for the analysis of the results of the agronomic characteristics of the plant, an analysis of variance was carried out and shown in Table 4. This result is due to the N contribution of the applied fertilizer, which influenced many biochemical reactions that favored leaf development and consequently favorably affected the translocation of nutrients to the bulb, thus obtaining higher yields when compared to the control sample. According to the analysis reported by NIAI-H (INIA, 2021a), it shows that the analysis of the ME obtained an adequate concentration of the macronutrient N.

3.4. Leaf analysis of beet

According to the results of the foliar analysis detailed in Table 5, it can be seen that as the effluent doses increased, the concentrations of K, P and S increased; however, the T5 treatment, which received the highest dosage, did not stand out in yield; it was the T4 treatment that stood out in yield. Therefore, it was analyzed that P, Mn and B influenced the yield and quality of the bulb. P is important in plant energy metabolism; it is part of AMP, ADP and ATP molecules as well as DNA and RNA and is involved in photosynthesis, respiration and starch synthesis. P is

Table 4. Treatments, doses, length, equatorial diameter, weight and commercial yield in the cultivation of beet.

Treatment	Dosage L/ha	Length of plant (cm)	Weight of one beet (g)	Diameter equatorial (cm)	Performance commercial (t/ha)
T4	5	43.36 a	205.38 a	6.69 a	23.968 a
T5	4	41.23 b	168.65 b	5.78 b	19.676 b
T3	3	38.65 c	157.32 c	5.32 c	18.135 c
T2	2	36.26 d	137.63 d	4.92 d	15.983 d
T1	0	33.56 e	101.56 e	4.46 e	12.367 e
CV		18.96	32.63	25.63	38.52

Note: Coefficient of variation (C.V): Means with the same letter in columns do not differ statistically ($p>0.05$).

Table 5. Leaf analysis of beet according to the doses (L/ha) of modified effluent.

Macro nutrients (g kg^{-1})	DT1	DT2	DT3	DT4	DT5	Values normal
K	2.25	2.71	3.19	3.31	3.49	2.00 - 6.00
N total	3.43	3.18	3.11	3.22	2.75	4.75 - 5.50
P	0.32	0.33	0.35	0.43	0.40	0.45 - 1.10
Ca	3.62	3.53	2.95	2.64	2.73	0.50 - 1.50
Mg	1.31	1.29	1.16	0.97	1.13	0.25 - 1.00
S	0.59	0.63	0.85	0.83	0.90	
Micro nutrients (mg kg^{-1})						
Fe	641	786	805	565	528	60.0 - 140
Mn	80.9	86.8	116	127	109	26.0 - 360
Cu	10.9	11.0	12.8	11.6	9.60	5.00 - 15.0
Zn	46.3	33.4	43.9	43.8	28.4	10.0 - 80.0
B	71.9	75.6	72.0	81.5	78.4	31.0 - 200
Mo	< 0.10	0.59	< 0.10	0.50	< 0.10	

Note: DT1 = 0, DT2 = 2, DT3 = 3, DT4 = 4 and DT5 = 5 are the fertilizer doses in L/200 L of water, applied to the 5 treatments, respectively.
Source: AGQ Labs (2022).

important in plant energy metabolism; it is part of AMP, ADP and ATP molecules as well as DNA and RNA and is involved in photosynthesis, respiration and starch synthesis (Mejia de Tafur and Menjivar, 2010). El manganeso (Mn) es un micronutriente importante para el crecimiento y desarrollo de las plantas y sustenta funciones metabólicas dentro de diferentes compartimentos celulares de las plantas (Alejandro et al., 2020). Considering that phosphorus is an indispensable element for plant growth, it has become necessary to look for alternatives that allow the efficient use of this nutrient by means of bacteria that can obtain soluble phosphate ions for the plant. (Corrales et al., 2014).

4. Conclusions

The neutralizing agent for the reduction of the free acidity of crude fish oil was replaced by using KOH instead of NaOH; on the other hand, a variation was also made by using NH_4OH to neutralize the acidity of the residual aqueous mixture, obtaining a product with the chemical characteristics of a fertilizer for foliar application. This variation in the neutralizing agent generated a fertilizer instead of a polluting effluent. The response of the beet plant to the application of the ME dosage showed improvements in the physical characteristics of all the products obtained in the T treatments as the ME dosage increased, a fact that was notorious in the T4 treatment, but this behavior was not observed in the T5 treatment; this atypical response in this treatment could be explained by the inadequate concentrations of nutrients, OM, N and by the alkaline tendency of the soil.

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