

Research article

# From agricultural residues to biofertilizers: preparation and characterization for use in hydroponics

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## Abstract

This article describes the procedures for collection, preparation and characterization of the agricultural residues (poultry, cattle manure and orange bagasse) and their mixtures (M1 - cattle manure and orange bagasse, M2 - poultry and orange bagasse, M3 - poultry and cattle manure, M4 - cattle manure, poultry and orange bagasse). The waste collection was made in the municipality of São Carlos aiming to give a benefit to the residues generated in the region. These residues were characterized as pH, humidity, total organic carbon (TOC) and NPK. Among these characterizations, the pH is the most worrisome. The low pH of the orange bagasse residue (pH = 3.65) may be a problem for crops. After the characterization of the residues, the same was done for the mixtures, namely: pH, electrical conductivity, saline index, lactic acid, TOC, macro and micronutrients and urease activity. In general, the M3 mixture was the more satisfactory concerning to the agronomic parameters evaluated.

**Keywords:** organic agriculture; biofertilizers; organic matter; organic waste.

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## Introduction

Currently, large amounts of chemical fertilizers and pesticides are being used by farmers to obtain a better yield of various crops [1]. However, the continuous and excessive use of these chemical fertilizers and pesticides changes the soil structure and can cause health problems to consumers [2].

An useful and cost-effective practice is the biofertilizers employment prepared from the aerobic or anaerobic digestion of organic materials to replace mineral fertilizers, mainly because of the growing demand for new production technologies, which should have a trade-off between lowering costs and improving quality for human and environmental health. In addition, the farmer himself can produce these products, saving of imported inputs and promotes improvements in environmental sanitation [3].

Biofertilizers are bioprotectors from a decomposition process of organic matter in water, resulting in a liquid waste. A biofertilizer acts as a fertilizer, stimulating insect repellent (increases the resistance of plants) and diseases controller.

According to Santos (2001)[4], biofertilizer is the name given to the liquid effluent obtained from aerobic or anaerobic fermentation of organic matter and water, while Medeiros and Lopes (2006)[3] define it as the final residue of the fermentation of organic compounds containing living cells or latent microorganisms (bacteria, yeasts, filamentous fungi and algae) and their metabolites.

The use of biofertilizers in hydroponic crops can be an environmental friendly alternative for the supply of nutrients, particularly for relatively short cycle crops such as lettuce. These compounds are rich in nutrients, in addition, they possess bioactive compounds [3], and almost all necessary macro and microelements for a healthy plant nutrition [5].

One of the problems in agriculture is the inadequate disposal of organic waste: in addition to the large area occupied, farmers lose nutrient rich material that could be a viable alternative for the replacement of fertilizers used in crops.

The region of São Carlos is characterized by intense agropastoral activity, in which the extensive cattle and feedlot cattle has important role. These activities generate organic wastes that can be used as feedstock for the production of biofertilizers, yielding final products with different chemical properties.

Cattle manure in natura is widely used as organic fertilizer, but the composting process makes it more stable and optimizes their fertilizing properties [6].

Orange is among the most produced and consumed fruit in the world, whose production exceeds 80 million tons/year. The orange peel is the main by-product of processing, corresponding to about 45% of the total weight of the fruit and can become a major problem for industry because decays very fast during storage.

The poultry production is now an activity in expansion and the region of São Carlos undergoes changes, including the implementation of poultry in small and medium-sized properties. The production of broilers, in this region, as well as the production of vegetables in greenhouses, are facts that put as productive pole and gave a new aspect to the small farmer. The use of poultry for biofertilizer production promotes both the environmental sanitation, and decrease adverse impacts on soil, water and air [7].

The aim of this paper was to prepare and characterize biofertilizers from organic residues produced in a large scale, in order to give a benefit to these products with respect to the hydroponic production of lettuce.

## Materials and Method

The experiments were mounted in plastic barrels in triplicate, in predetermined volumes, to which the residues were mixed in their proportions corresponding to each treatment. The mixtures were: cattle manure + orange bagasse (M1); poultry + orange bagasse (M2); cattle manure + poultry (M3) and cattle manure + poultry + orange bagasse (M4). The residues were mixed in equal amounts (v/v or v/v/v). For the biofertilizer, 20% by volume of each mixture and 80% of water (40 liters = 32 liters of water + 8 liters of the mixture containing the residues) were used. Each mixture was homogenized twice a day to avoid rotting and left to rest without contact with sun light or rain. Samples were collected for characterization on days 1, 15, 30, 40, 50 and 60 for further comparisons. According to MEDEIROS et al. (2003) [8], the biofertilizer was ready for use from the 30th day, yet the collections continued to evaluate stabilization. The pH, electric conductivity, salt index, lactic acid, total carbon content, the nutrients NPK and Ca, Mg, Cu, Fe, Mn and Zn and urease activity were determined in order to evaluate the dynamics of agricultural residues transformation in biofertilizers.

The pH of the biofertilizers was evaluated with the aid of a probe, which allows the reading of pH in situ, i.e., at the time of collection. The determination of the electrical conductivity (EC) was performed by means of conductivity in the collected sample itself. The salt index (SI) was calculated based on the electrical conductivity of the biofertilizers taking into account the conductivity values of  $\text{NaNO}_3$  according to the formula:

$$SI(\%) = \frac{100 \times EC_{\text{biofertilizers}}}{EC_{\text{NaNO}_3}}$$

Lactic acid in this experiment was determined according to the methodology described by Adolfo Lutz Institute (Brazil, 2005)[9] for milk, which estimates the lactic acid indirectly through the titratable acidity. This methodology consists in neutralizing the sample via titration in the presence of a predetermined alkali solution using phenolphthalein as indicator in alcoholic solution.

The total organic carbon was determined in an elemental carbon analyzer (TOC-VCPH) coupled to the solid samples module, SSM-5000A, SHIMADZU (with combustion detector), TOC determination was instrumental [10].

For the determination of N, P, K, Ca, Mg, Cu, Fe, Mn and Zn, the biofertilizers were first digested in a closed microwave oven (SpeedWave four - microwave digestion system with Built-in, non-contact temperature and pressure measurement). Nitrogen was determined as the total Kjeldahl nitrogen (TKN) by Hach® method 399 and phosphorus by Hach® method 480 [11]. Potassium and others nutrients were analyzed by atomic absorption spectrometry.

To determine urease activity, 0.2 mL of toluene, 9 mL of THAM buffer pH 9.00 and 1 mL of  $0.02 \text{ mol L}^{-1}$  urea were added to 0.5 g of the Freeze-dried biofertilizer and then the mixture was incubated at  $37^\circ\text{C}$  for 2 h before the volume was made up to 50 mL with  $\text{KCl-AgSO}_4$ . The ammonium released was determined using steam distillation [12].

All determinations were performed in triplicate.

## Results and Discussion

### Characteristics of the residues

For comparison purposes, the residues in natura were also characterized concerning to pH, humidity, total organic carbon and NPK (Table 1).

**Table 1** - Attributes the in natura residues before fermentation.

Attributes	Cattle manure	Poultry	Orange bagasse
pH in CaCl <sub>2</sub>	10.27 ± 0.04	8.48 ± 0.0	3.65 ± 0.02
Humidity (%)	9.53 ± 0.06	11.90 ± 0.09	9.07 ± 0.07
TOC (mg kg <sup>-1</sup> )	48,626.67 ± 932.28	57,256.67 ± 930.07	70,863.33 ± 2.000
N (mg kg <sup>-1</sup> )	423.33 ± 10.41	807.5 ± 48.8	316.5 ± 40
P (mg kg <sup>-1</sup> )	4,056.39 ± 65.22	4,905.33 ± 27.98	3,825.74 ± 60
K (mg kg <sup>-1</sup> )	3.5 ± 0.06	5.0 ± 0.01	2.0 ± 0.01

As we can see, each residue presented specific characteristics. Cattle manure and poultry present a basic character due to the constituents of their structure. Already the orange bagasse is acid, this was already expected because the presence of citric acid.

The values of nitrogen, potassium and phosphorus are very important because these nutrients are essential for the growth of crops. We can observe from the Table 1 that the poultry presents the highest values for these macronutrients, this does not mean that this residue was the best, because during the fermentation process they may have been mineralized making them unavailable. Once again, it is worth remembering that these attributes change when the mixtures are performed and consequently the fermentation, since one will interfere in the composition of the other throughout the process.

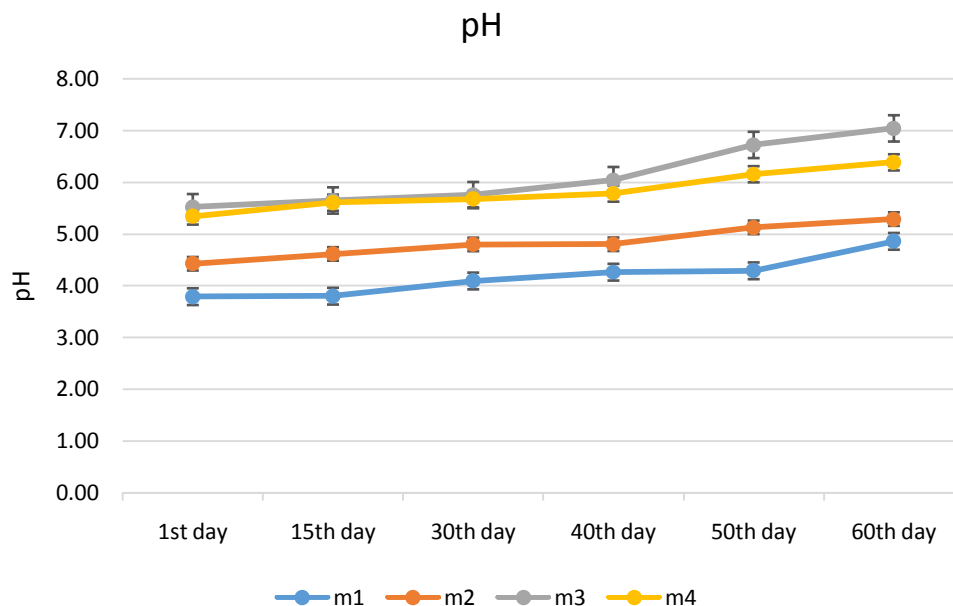
Next, the results will be discussed for the biofertilizers formed.

### Hydrogen potential (pH)

As can be seen in Figure 1, mixtures containing orange bagasse (M1 and M2) in their composition practically maintained their acidic values of pH, going from 3.76 to 4.87 in M1, and from 4.41 to 5.24 in M2. This may be due to the presence of the citric acid in the residues, which hinders or even prevents the process, leaving the acidic mixture throughout the period. For these mixtures, these values are detrimental to hydroponic crops because they prevent the absorption of nutrients, since this occurs in the pH range of 5.5 to 7.0.

In M3, where the orange bagasse was not present, it can be seen that the initial pH value (pH = 5.48) already starts near the ideal range of nutrient uptake reaching the end of the process near neutrality with a pH equal to 7.01, showing that in relation to pH, M3 was the best mixture among all.

M4 although it still has 1/3 of orange bagasse on its composition, also contained poultry, which helped the mixture to ferment and reaching pH values close to 6.45.

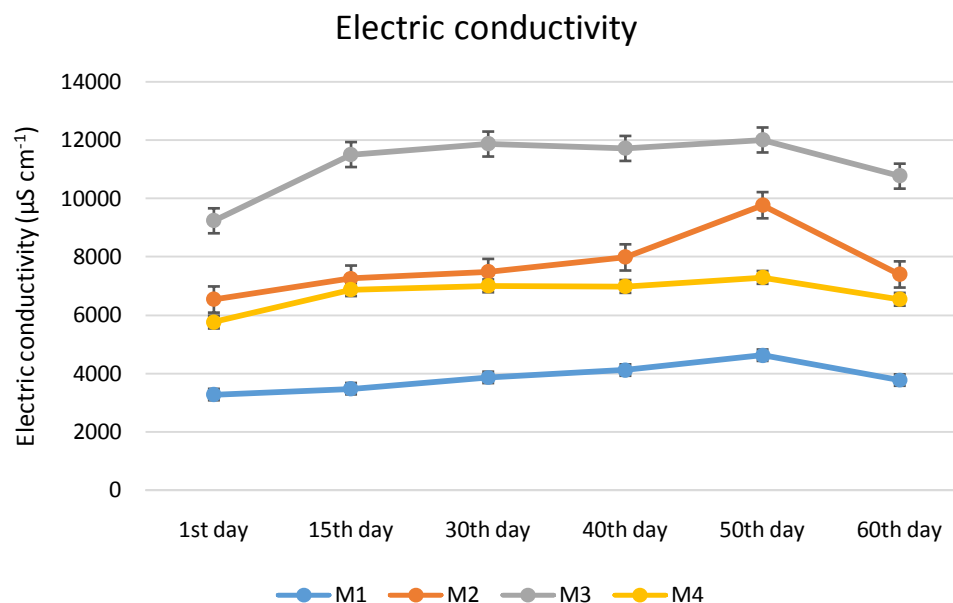


**Figure 1** - pH values of the samples in the mixtures M1, M2, M3 and M4 during the fermentation process. M1 - cattle manure and orange bagasse; M2 - poultry and orange bagasse; M3 - poultry and cattle manure; M4 - cattle manure, poultry and orange bagasse.

### Electric conductivity (EC)

The determination of the EC of the biofertilizers was done to estimate the total ion content in solution, based on the principle that the resistance to the passage of electric current, under standard conditions, decreases proportionally with the increase in the concentration of salts, that is, the greater the presence of nutrients, which are conductive in solution, the higher the conductivity.

As observed in the Figura 2, the electric conductivity increased until 50<sup>th</sup> day, showing that until this stage the nutrients are becoming more available. In addition to this, we can see that the last collection had lower conductivity values than previous ones in all mixtures, this is an indicative that the process can be interrupted. Among the mixtures, M3 was the one that presented the highest conductivity values in all processes, it being understood that this mixture may be more nutritious in relation to others.



**Figure 2** - Values of the electrical conductivity ( $\mu\text{S cm}^{-1}$ ) of the samples in the mixtures during the fermentation process.

M1 - cattle manure and orange bagasse; M2 - poultry and orange bagasse; M3 - poultry and cattle manure; M4 - cattle manure, poultry and orange bagasse.

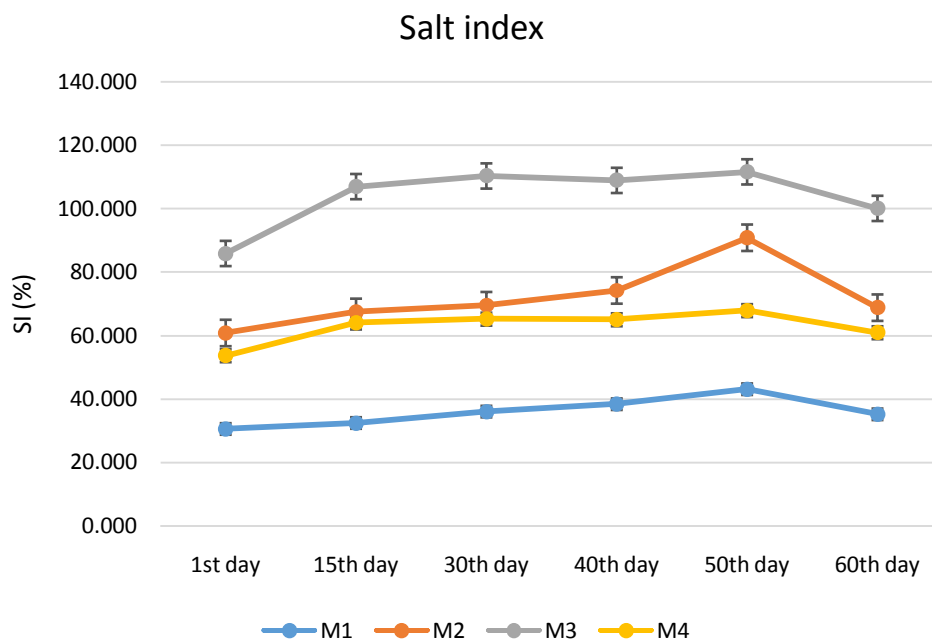
### Salt index (SI)

This is related to the electric conductivity of biofertilizers. Salt index shows an estimate of the available amount of salts in the soluble medium. A mixture with more available salts is expected to be more nutritious.

As can be seen in Figure 3, it appears that the salt index behavior did not have many variations throughout the collections. What we can perceive is that among the mixtures, once again M3 presents higher values of SI. From 15<sup>th</sup> day to 60<sup>th</sup> day, M3 presented values even larger than  $\text{NaNO}_3$  (material used as comparison standard), reaching its highest value on the 50<sup>th</sup> day ( $\text{SI} = 111.63 \pm 0.12\%$ ), showing that poultry and cattle manure contribute to the nutritional value of this mixture.

We can also observe that the mixture M2 had the second best SI values. This may be related to the fact that M2 and M3 present 50% of poultry, a material rich in nutrients showing a tendency of this residue to provide nutrients in the medium throughout the process.

Once again, with M1 the worst results were obtained. There is practically no variation throughout the process. We can relate this to the fact that orange bagasse along with cattle manure did neither contribute for a mixture capable of fermenting nor make the necessary nutrients available for the crops. The same can be observed for M4, having little variation of SI values, which means that these nutrients practically did not become available throughout the fermentation process, which makes the use of this mixture unfeasible.

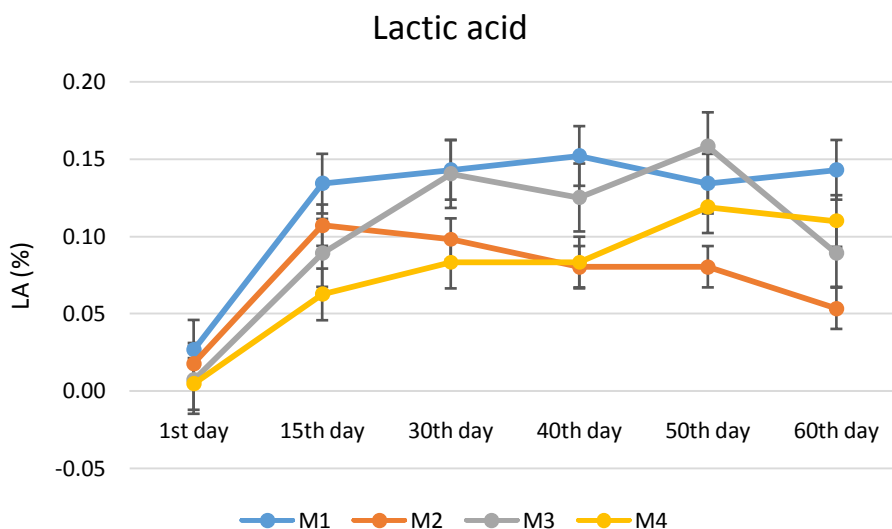


**Figure 3** - Values of the salt index content of the samples in the mixtures during the fermentation process. M1 - cattle manure and orange bagasse; M2 - poultry and orange bagasse; M3 - poultry and cattle manure; M4 - cattle manure, poultry and orange bagasse.

### Lactic acid (LA)

Lactic acid is formed throughout the fermentation process, so an increase in the percentage of lactic acid is expected as the fermentation develops.

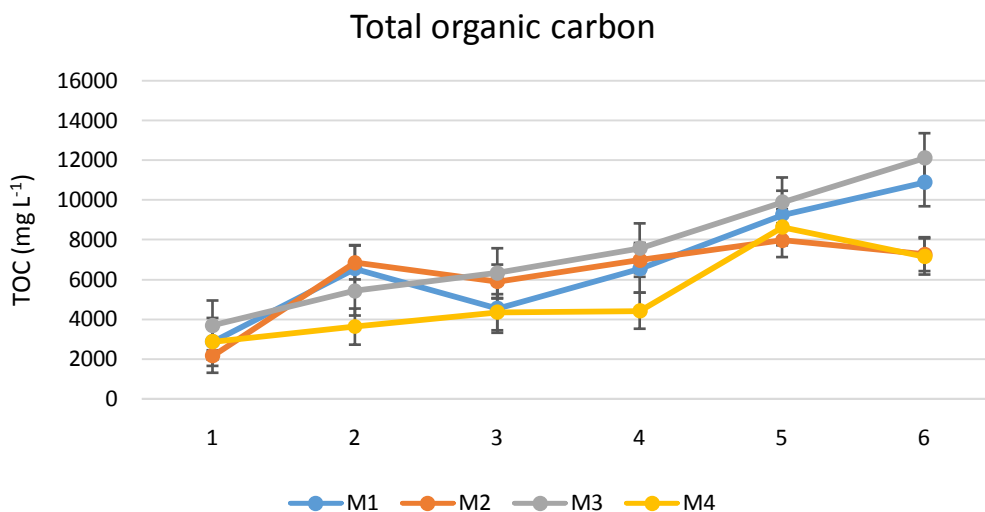
For lactic acid values shown in Figure 4, there was a large difference when compared the first day with the others, as expected. While the collection made on the first day of preparation was almost zero for all mixtures, from the second collection made on 15<sup>th</sup> day of fermentation, values increased significantly reaching close to 0.16%. This attribute is directly related to the fermentation of biofertilizers, that is, while LA values are increasing, this shows a tendency that the fermentation is occurring, and the decrease of this attribute may be related to a decrease of the fermentation process or even its inhibition.



**Figure 4** - lactic acid values of the samples in the mixtures during the fermentation process.  
 M1 - cattle manure and orange bagasse; M2 - poultry and orange bagasse; M3 - poultry and cattle manure; M4 - cattle manure, poultry and orange bagasse.

**Total organic carbon (TOC)**

The determinations of total organic carbon were made to predict the organic matter content present in the biofertilizers, which is directly linked to the fertility of the product (Figure 5). M3 presented a continuous increase in the total organic carbon content during the fermentation process.



**Figure 5** - Values of the total organic carbon content of the prepared mixtures.  
 M1 - cattle manure and orange bagasse; M2 - poultry and orange bagasse; M3 - poultry and cattle manure; M4 - cattle manure, poultry and orange bagasse.



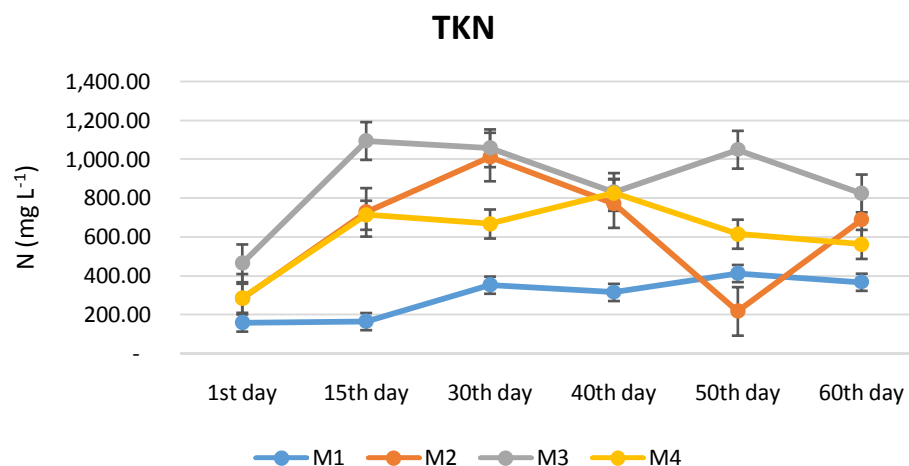
### Macro and micronutrients

Macronutrients are of fundamental importance in the vegetative growth. Nitrogen, phosphorus and potassium possess an important nutritional role in the plant growth.

The nitrogen data in Figure 6 show that for all the mixtures there was an increase of the total Kjeldahl nitrogen concentration in the first 30 days of the fermentation process. It means that during this period there was formation of organic nitrogen, being that in M2 and M3 this increase was more expressive, showing that poultry is a nitrogen rich residue able to meet the needs of plants.

It can be observed that from the 30<sup>th</sup> day, except for M1, the nitrogen concentrations began to decrease, indicating a possible mineralization.

The Ministry of Agriculture, Livestock and Food supply in its normative instruction n°25 of 2009 reports that for NPK in nutrient solution for hydroponics there is no maximum or minimum value indicated, only requires that the product must contain in its label the specifications of each nutrient.

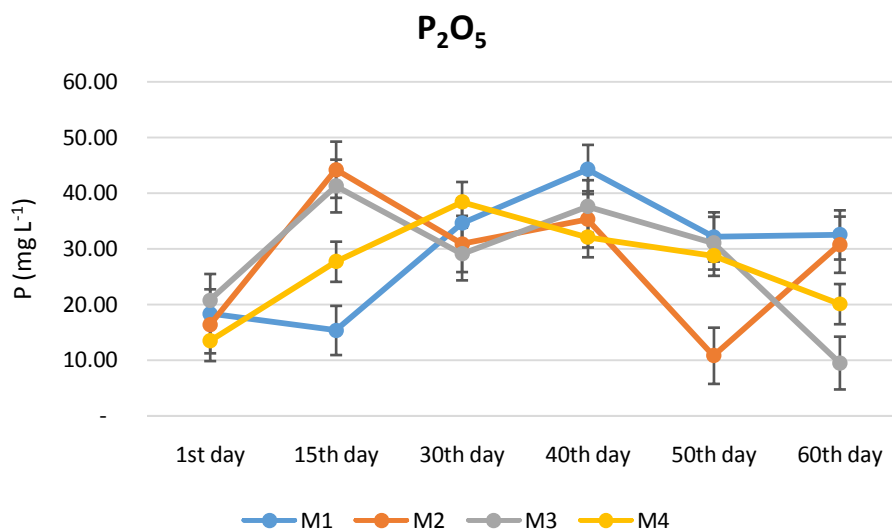


**Figure 6** – Total Kjeldahl Nitrogen values in mg L<sup>-1</sup> for biofertilizers prepared with organic residue. M1 - cattle manure and orange bagasse; M2 - poultry and orange bagasse; M3 - poultry and cattle manure; M4 - cattle manure, poultry and orange bagasse.

The lack of nitrogen in the initial phase of the vegetative growth can delay the growth and, consequently, the production, besides making the leaves with a pale green coloration.

Phosphorus is one of the direct agents of the formation of chlorophyll and increases the root development, giving the plant greater capacity to absorb the fertile elements of the soil.

The phosphorus data do not have a trend, being random throughout the process. What can be said is that for M1, M2 and M4 the final concentration of phosphorus on the last day was higher when compared to the first day, showing that even with the concentration variations throughout the process, at the end, phosphorus was more available than at the beginning of the process. For M3, this tendency to make phosphorus more available was not seen, since the concentration decreases at the end of the fermentation. This is another indicative that the process can be interrupted at the 50<sup>th</sup> day at the most.

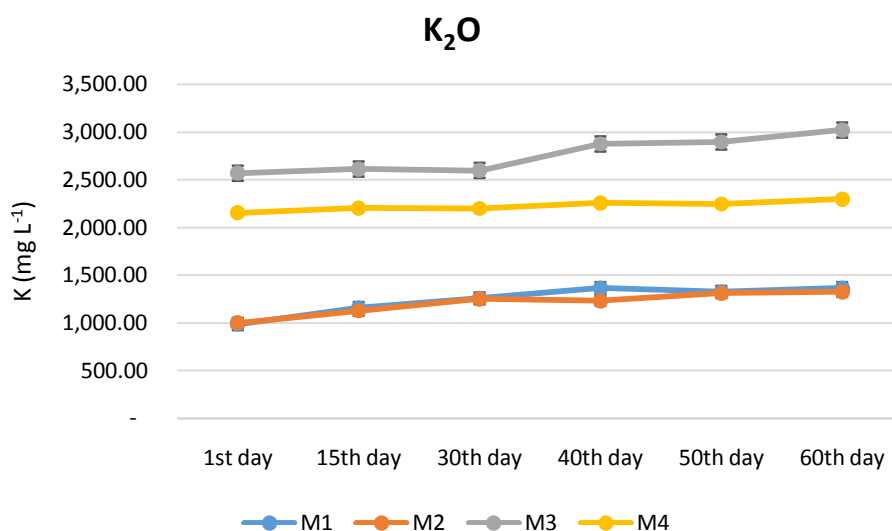


**Figure 7** - Phosphorus values in phosphate form in mg L<sup>-1</sup> present in biofertilizers.

M1 - cattle manure and orange bagasse; M2 - poultry and orange bagasse; M3 - poultry and cattle manure; M4 - cattle manure, poultry and orange bagasse.

Differently from the nitrogen and phosphorus data, the potassium (Figure 8) showed a gradual increase throughout the monitored time, with the highest concentrations in M3 in the whole process. We can say that M1 and M2 practically had the same behavior all the time. For all the mixtures, the concentration remained the same practically the whole fermentative process.

It is worth remembering that the variation between the values present earlier is because no situation was forced, although everything was done in protected environment, all the preparation was in real scale, without external interferences.

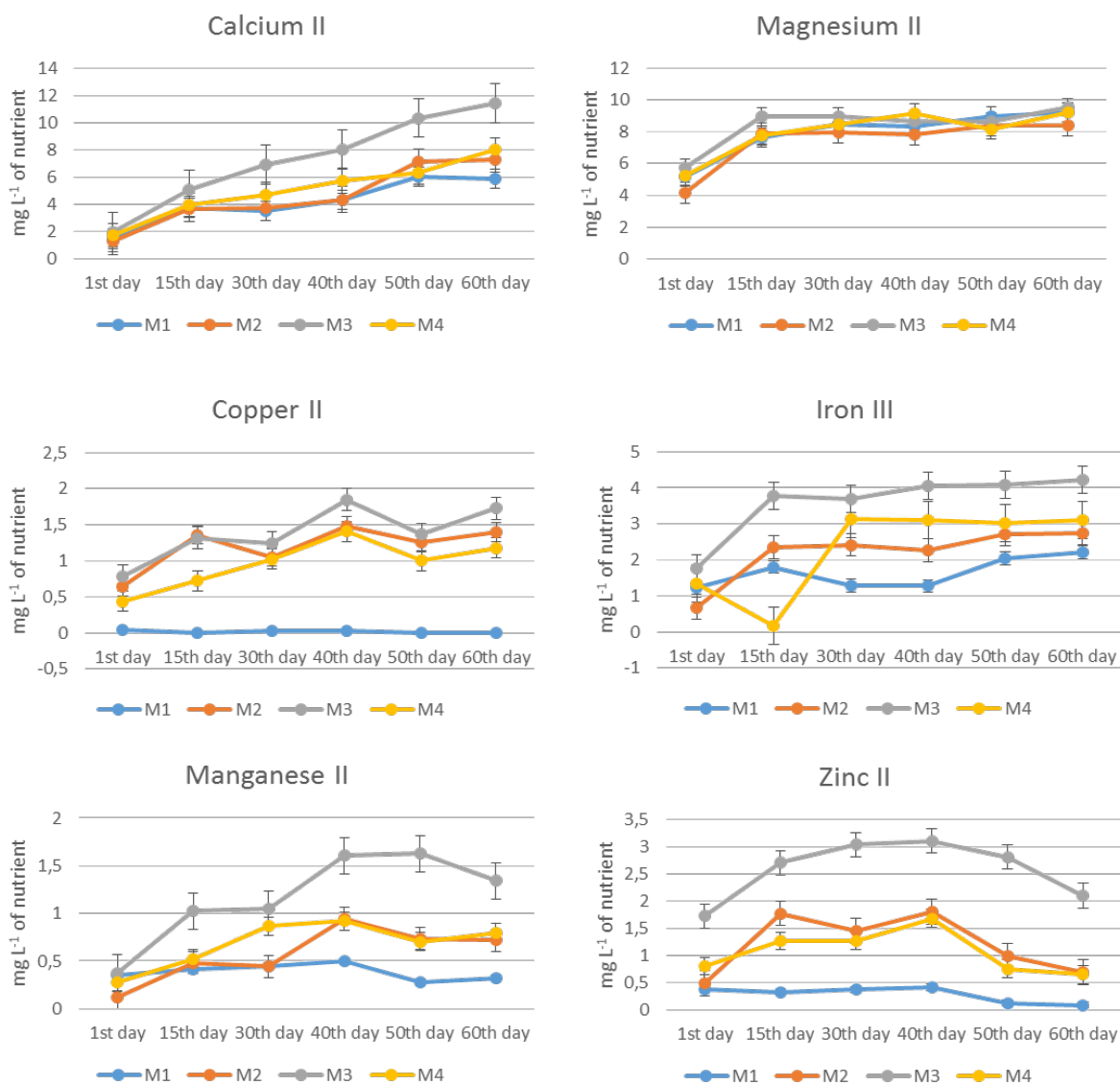


**Figure 8** - Potassium values in mg L<sup>-1</sup> present in biofertilizers.

M1 - cattle manure and orange bagasse; M2 - poultry and orange bagasse; M3 - poultry and cattle manure; M4 - cattle manure, poultry and orange bagasse.

For Ca, Mg, Cu, Fe, Mn and Zn, all mixtures had an increase in their availability throughout the fermentation process. This is good for the nutritional character of biofertilizers, showing that fermentation helps in releasing the nutrients making them available.

With the results shown in Figure 9, we can observe that for all nutrients, M3 presents the highest nutritional availability at the end of the process. By showing for these attributes, M3 tends to be more nutritious in relation to others. Calcium and magnesium, also known as exchangeable bases, in all mixtures were the most commonly found nutrients. This is a positive factor for the nutritional character of biofertilizers, since the two nutrients are an indispensable nutritive cations for the growth of plants.



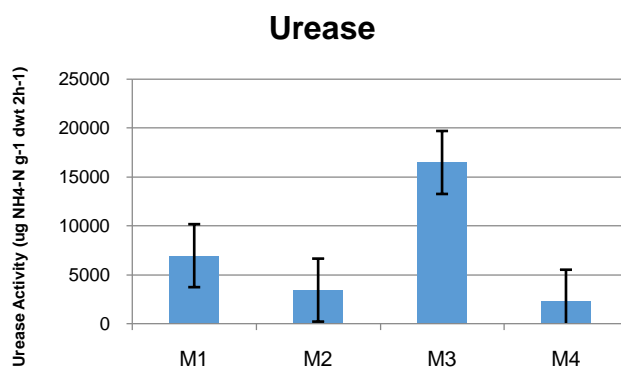
**Figure 9** - Graphs of micronutrients comparing M1, M2, M3 and M4.

M1 - cattle manure and orange bagasse; M2 - poultry and orange bagasse; M3 - poultry and cattle manure; M4 - cattle manure, poultry and orange bagasse.

Unlike other nutrients, manganese and zinc do not show significant results in mixtures, ie, these elements do not follow a trend seen in the other micronutrients. This may be due to the interference of the other elements in their availability, and a likely competition for the active sites, with manganese and zinc cations less interacting with the biofertilizer in question.

### Urease

Urease assay is used in predicting the release of nitrogen from organic amendments when added to the soil. The urease activity was highest in M3 (Figure 10).



**Figure 10** - Values of the urease content of the samples in the barrels during the fermentation process. M1 - cattle manure and orange bagasse; M2 - poultry and orange bagasse; M3 - poultry and cattle manure; M4 - cattle manure, poultry and orange bagasse.

This is an indication of the disappearance of ammonium, which can act as inhibitor. The biofertilizers in M1 and M3 showed higher urease activity suggesting cattle manure has a favored urease activity or the degradation of cattle manure gave rise to the formation of nitrogen substrate susceptible to use by this enzyme [13][14]. According to the data of TKN (Figure 6), with can suppose that the contribution of organic nitrogen is the most important factor in TKN values.

### **Conclusion**

The period of formation of the biofertilizers influenced all the analyzed attributes.

The biofertilizers containing poultry (M2, M3 and M4) presented the highest nutrient values as well as the best pH and electric conductivity.

In all analyzed attributes, M3 was the mixture that had better results when compared to others, being an indicative that this mixture will give a better agronomic value for the hydroponic lettuce cultivation.

In order to prove the best biofertilizer in relation to the productivity of lettuce hydroponic planting, the next step of this study will contemplate the application of the mixtures in the culture mentioned.

Thus, we recommend a careful evaluation of the adopted agricultural practice (hydroponics), in order to monitor the growth of lettuces and thus evaluate the nutritional balance of each mixture.

## Acknowledgement

The authors would like to acknowledge to FAPESP (São Paulo Research Foundation), process number 2016/11658-5; CNPq (National Council for Scientific and Technological Development), process number 306715/2013-9 and CAPES for financial support.

## References

- [1] JOSHI, R.; VIG, A.P. Effect of Vermicompost on Growth, Yield and Quality of Tomato (*Lycopersicum esculentum* L), African J. Basic & Appl. Sci. v. 2, n. 3-4, 2010, p. 117-123.
- [2] ASGHAR, H.N.; ISHAQ, M.; ZAHIR, Z.A.; KHALID, M.; ARSHAD, M. Response of radish to integrated use of nitrogen fertilizer and recycled organic waste. Pak. J. Bot. v. 38, n. 3, 2006, p. 691-700.
- [3] MEDEIROS, M.B. de; LOPES, J. de S. Biofertilizantes líquidos e sustentabilidade agrícola. Bahia Agrícola, v.7, 2006 p. 24-26.
- [4] SANTOS, A. C. V. A ação múltipla do biofertilizante líquido como ferti fitoprotetor em lavouras comerciais. In: Hein, M. (org.). ENCONTRO DE PROCESSOS DE PROTEÇÃO DE PLANTAS: CONTROLE ECOLÓGICO DE PRAGAS E DOENÇAS, I. 2001, Botucatu. Livro de Resumos... Botucatu: Agroecológica, 2001. p.91-96.
- [5] SILVA, A.F.; PINTO, J.M.; FRANÇA, C. R. R. S.; FERNANDES, S. C.; GOMES, T. C. de A; SILVA, M. S. L. da; MATOS, A. N. B. Preparo e uso de Biofertilizantes Líquidos. Comunicado Técnico da Embrapa SemiÁrido, 2007, 130 p.
- [6] FIALHO, L. L. Caracterização da matéria orgânica em processo de compostagem por métodos convencionais e espectroscópicos. 2007. 170 f. Tese (Doutorado em Ciências, Química Analítica) – Instituto de Química de São Carlos, Universidade de São Paulo, São Carlos, 2007.
- [7] NASCIMENTO, G. A. Z. Gestão de resíduos em propriedade rural: utilização de resíduos avícolas para a produção de energia e biofertilizante. 2011. 113 f. Dissertação (Mestrado) – Escola de engenharia Mauá do Centro universitário do Instituto Mauá de Tecnologia, São Caetano do Sul, 2011.
- [8] MEDEIROS, M. B.; WANDERLEY, P. A.; WANDERLEY, M. J. A. Biofertilizantes líquidos. Revista Biotecnologia Ciência e Desenvolvimento, v. 31, 2003, p. 38-44.
- [9] Instituto Adolfo Lutz (São Paulo). Métodos físico-químicos para análise de alimentos/coordenadores Odair Zenebon, Neus Sadocco Pascuet e Paulo Tiglea -- São Paulo: Instituto Adolfo Lutz, 2008 p. 1020.
- [10] ZOZOLOTTO, T. C.; Zozolotto, H. C.; Silva, P. R. D.; Landgraf, M. D.; REZENDE, M. O. O. Validação de metodologia para determinação do extrato húmico total em matrizes sólidas. ANALYTICA, v. 61, p. 58-67, 2012.
- [11] Dores-SILVA, P. R.; Silva, B. M.; ZOZOLOTTO, T. C.; LANDGRAF, M. D.; Rezende, M. O. O. Understanding the vermicompost process in sewage sludge: a humic fraction study. International Journal of Agriculture and Forestry, v. 4 (2), p. 94-99, 2014.
- [12] Delgado, A., Solera del Río, R., Sales, D., J. L. García-Morales.. Study of the co-composting process of municipal solid waste and sewage sludge: stability and maturity. *Organic Waste Treatments: Safety Implications*. 2004, p. 257-260.
- [13] Kandler, E., Poll, C., Frankenberger, W. T. and M. Ali Tabatabai. Nitrogen enzyme cycles. In: R. P. Dick, editor, Soil Enzymology. Agron. Monogr. 48. ASA and SSSA, Madison, WI. 2011, p. 211-245.

[14] Ross, M., García, C. and T. Hernández. A full-scale study of treatment of pig slurry by composting: Kinetic changes in chemical and microbial properties. *Waste Management* 26: 2006, p. 1108–1118.