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Comparative Study under Controlled Conditions of the Nitrogen Fertiliser Value of the Two Residual Organic Products (ROPS) Originating from Distillation Activities of Clove Leaf Essences: Case of 4-month-old leaf compost and about 40-year-old leaf mold

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ABSTRACT

This study brought out the nitrogen fertilizing value of (2) ROPs originating from the activity of distilling essential oil from cloves leaves. Indeed, many deposits of waste related to the activity of distilling essence of clove leaves are identified in all the clove areas of the Analanjirofo region (Madagascar) but unfortunately this waste remains untapped or even undervalued until now. Some of them are very old, sometimes reaching the age of 40 or even over. The study we carried out shows that these original PROs of distillation activity have a significant nitrogen fertilizer value, possibly due to the age and stability of their organic matter. In addition, the 04 months old leaves compost shows a fairly satisfactory nitrogen fertilization value.

Key words: leaf mold, PRO, fertilizer value, incubation, essential oil of clove leaves

1. INTRODUCTION

Organic waste products (ROP) are an important source of nitrogen fertilisation, but their nitrogen value is still difficult to accurately assess [1].

Indeed, the nitrogen contained in the products is present in the mineral form and in the form of different organic molecules. This nitrogen is therefore available for crops in very variable times, from a few days for mineral forms to several years (or even several decades) for some organic forms. Knowledge of the nitrogen value of composts is a major demand of farmers [2].

Note that nitrogen value means the ability of composts to provide nitrogen to plants.

There are in fact three types of experiments measuring the nitrogen value of the ROPs in this case:

- ✓ Experiments in the presence of plants in the field make it possible to assess the effects of inputs from PROs on soil nitrogen dynamics, bioavailability and plant growth under conditions similar to agricultural practices [3,4,5,6,7,8,9],
- ✓ Experiments with plants and pots also show the effects on plants [10,11,12,13],
- ✓ Laboratory experiments without plants with mixtures containing soil and PRO placed in incubation under controlled conditions [8,14,15,16,17,18,19].

Certainly, there are a number of models simulating nitrogen transformations in the soil, usually together with carbon. The models NCSOIL, CERES, CANTIS for example allow to simulate the nitrogen and carbon turnover from several compartments [17,20]. Models such as LIXIM [21,22] and DAISY [23] are used to simulate nitrogen mineralization and leaching.

Isotopically labelled nitrogen (N^{15}) is also used in a number of studies on soil nitrogen dynamics.

Such a tracer makes it possible to estimate the crude quantities of nitrogen immobilized and mineralized by biomass, or assimilated by plants [24,25,26,27] or to study the*« primingeffect »* linked to the incorporation into the soil of an exogenous material [28].
The purpose of this study is to determine in controlled incubation the availability of nitrogen in the short and medium term of the two ROPs resulting from the distillation activity of clove leaf essence, namely: 4-month old bush still leaves composts from Iazafo's composting platform composting and leaf loam of about 40-year old leaves from residues of essential oil distillation leaves of cloves collected at the Ambodimanga II distillation site, Vavatenina district Analanjirofo region, country Madagascar.
Our choice is indeed on this type of ROP of origin of the distillation activity because the Analanjirofo Region (Madagascar) has many sites of distillation of essence of leavesin all the clover zones of the said region. These distillation plants generate large quantities of leaf waste every day and this during almost the whole year. Moreover, astronomical quantities of this leaf waste remain untapped (not recovered) since the introduction in Madagascar of the first distillation still of leaf essence in 1911 [29,30].

2. MATERIALS AND METHODS

2.1. Organic Waste Products

The two ROPs objectives of this study are:
- a compost of waste from four-month old bush still leaves supplied by Iazafo Compost (see Photo 2 below). These leaf residues are the fresh discharges resulting from the distillation operation of essential oils from the leaves of the clovers and composted under controlled conditions on the composting platform of the Company Iazafo Compost for four months,
- a loam of leaves buried for about 40 years and collected on the distillation site of Ambodimanga II, municipality of Maromitety, Vavatenina district , Analanjirofo region (Madagascar).

This leaf loam is the fruit of the natural decomposition without human intervention of leaf residues discharged by operators-distillers around the distillation site after each distillation operation and those for about forty years or more for some distillation sites. Thanks to the pedoclimatic conditions of the Analanjirofo Region (hot and humid), these leaves residues decompose spontaneously and over time, eventually forming soil rich in stable and moistened organic matter.
Here it is necessary to specify that this is the result of survey and interview with notables (tangalamena) and residents and especially owners of stills from the Ambodimanga II site, who have provided us with valuable information on the age of these soils formed around the Ambodimanga II site (see photo 1 below). According to them, this distillation site in Ambodimanga has been operational since 1979, an estimated 40 years of existence in 2020.

Photo 1: Sample collection of Ambodimanga II leaf loam approximately 40 years old (personal source)

Photo 2: 4-month old mature leaf compost from the Iazafo composting platform (Source: Iazafo compost, 2020)

2.2. Agronomic Analysis

In order to allow us to better appreciate the agronomic value of each ROP, more precisely the nitrogen content, we proceed to the classical agronomic characterization of each ROP including the determination: of the percentage of dry matter (% DM), total organic matter (TOM), total organic carbon (TOC), total nitrogen (total N), organic nitrogen (Norg), ammoniacal nitrogen ($N\text{-}NH_4^+$), nitric nitrogen ($N\text{-}NO_3^-$), pH, electrical conductivity (EC), C/N ratio
-determination of dry matter (DM) is carried out according to the standard protocol (NF ISO 11465, 1994): drying of a wet sample at 105°C to constant weight. The difference in mass before and after drying serves as a measure for the dry matter and water content [31],

-the total organic matter content (OM in % of DM) was determined by the mass loss when the sample was calcined at 550°C for 2 hours in a furnace (European standard EN 15935,2013) [32],
-total organic carbon will be determined by wet or sulfochromic oxidation (NF ISO 14235, 1998) [33],
-total nitrogen by the Kjeldhal method (NTK) (EN 13342, October 2000) [34],
-the determination of the organic nitrogen fraction contained in each ROP shall be made by subtracting the ammoniacal nitrogen concentration from the total Kjeldhal nitrogen concentration in the sample,
-the measurement of pH is carried out on aqueous suspensions according to afnor NF ISO 10390 of November 2005 and the measurement of pH (0,1 pH unit) is carried out directly by reading on a combined electrode pH meter [35],
-Specific electrical conductivity (EC) is measured on a 1/5 aqueous extract (compost mass/solution volume) at a temperature of 20 1°C (NF ISO 11265) [36]
-ammoniacal and nitric nitrogen by extraction with potassium chloride solution (KCl 1M) and then determined by colorimetric method (ISO 14256-1, 2003; ISO 14256-2, 2005) [37,38]. The absorbance is measured at 543nm for nitrates by the modified Griess-Ilosvay reaction [39,40] while the ammonium absorbance is measured at 630 nm by the alkaline Berthelot reaction [39,40,41].
- the C/N ratio calculated from TOC measured by sulfochromic oxidation and total nitrogen obtained by the Kjeldhal method (N-NTK),

2.3. Incubation test

For a ROP, two nitrogen effects are distinguished: (i) nitrogen in mineral form ($NH_4^+ + NO_3^-$) directly available to the plant (immediate supply), (ii) nitrogen in organic form available in the medium and short term (available during the first 12 months of its application) even in the long term depending on the dynamics of the mineralization of the organic matter of the ROP.

The dynamics of nitrogen supply depends on the type of ROP, particularly on the nature of the initialcomposition of the incoming waste in the composition of the ROPs.

The knowledge of the kinetics of short-term mineralization of organic nitrogen of ROPs through the study of incubation under controlled conditions in a laboratory makes it possible to predict the potential mineralization of a PRO particularly within 12 months following the intake (FD U 44-163) [42]. This standard, or more accurately this booklet of documentation, defines the potential for carbon and nitrogen mineralization of ROP. The experimental protocol consists in placing in controlled conditions (humidity pF2.5, temperature 28°C, duration of 91 days) the soil mixture + ROP. The ROP must be dried at 38°C+/-2°C, crushed and sieved (all must pass through a 1 mm sieve). The incubation soil is a loamy or sandy agricultural soil (clay 25%, pHwater7.3, $CaCO_3$ 0.2%, Corg 10g/kg, Nminéral 35 mg/kg DM). The sample of this soil will then be air-dried and sieved to 4 mm. Each sample is then composed of an equivalent of 25 g of dry soil at 105°C, and a mass of PRO such that the organic carbon input represents 0.2% of the dry soil mass (i.e. 2,000 mg of carbon/kg of dry earth + KNO_3 35 mgN/kg dry earth).

The measurement of mineral nitrogen, $NO_3^- + NH_4^+$, is done by extraction KCl 7 times after 0, 7, 14, 28, 49, 70 and 91 days of incubation at 28°C and then determined by colorimetric method [37,38] using the Berthelot method in alkaline medium for the determination of $N\text{-}NH_4^+$ and the Griess and Ilossay method for the determination of $N\text{-}NO_3$ [43].

From these measurements, the percentage of nitrogen mineralization of each product is deduced.

Net nitrogen mineralization is calculated by subtracting the amount of mineral nitrogen from the control soil at each date. The nitrogen mineralization coefficient may be expressed as a % of nitrogen, or Maximum Coefficient of Mineralization of Nitrogen:

$$\text{CMN (\%Norg)} = \frac{\max(\text{NNO}_3{}^- + \text{NNH}_4{}^+)}{\text{N organic brought}} \text{X100}$$

With: $-NO_3^-$: nitric nitrogen; $-NNH_4^+$: ammoniacal nitrogen

3. RESULTS AND DISCUSSIONS

3.1. Agronomic Analysis

The following table summarizes the results of physico-chemical analysis carried out on these two PROs:

Table -1 Main physico-chemical characteristics of the two ROPs

ROPs	Dry matter (%RM)	pH water	Electrical conductivity ($mS.cm^{-1}$ to 20°C)	Organic matter (% of DM)	Total Organic Carbon (% of DM)	Nitrogen kjeldahl (% of RM)	Organic nitrogen (% ofRM)	Ration C/N	Nitric nitrogen (g/kg of RM)	Ammoniacal nitrogen (g/kg of RM)
4 month old leaf compost	57.8	7	2	88.02	42.71	1.731	1.687	14.26	1.89	0.44
40-year-old leaf mold	61.74	6.1	691.10^{-6}	74.84	42.62	1.87	1.86	14.11	69.10^{-3}	$<10.10^{-3}$

Faced with compost of leaves only 4 months old, leaf soil seems aprioris richer in organic nitrogen. This situation is probably related to a better reorganization of the mineral nitrogen (ammoniacal nitrogen more precisely) during the natural process of much longer maturation of the soil (Xavier S., Personal communication). This finding also corroborates the result published by Equiterre (2009) [44].
Moreover, here, the loam of leaves aged 40 years is almost devoid of ammoniacal nitrogen. Leaf loam is obviously low in nitric nitrogen (69.10^{-3} g/kg RM) because over time, this loam has obviously been exposed to excessive leaching phenomena, especially by rainwater. This is why its electrical conductivity value is extremely low (691.10^{-6} $mS.cm^{-1}$). Another possible explanation is the existence of anaerobic conditions, which would lead the microflora to use NO_3^- as an oxygen source [45]. However, it is possible that during the formation of humic substances, significant amounts of salts could potentially attach to the stabilized OM [45] and thus cause the electrical conductivity (EC) of the composts to decrease. In addition, the pH of this leaf compost is slightly acidic (pH=6.1) compared to the pH of the 4-month-old leaf compost (pH=7). This reflects anaerobic conditions during the process of its biological transformation, leading to excessive acidity of the soil obtained [45]. Note that these leaf loams have not undergone any reversal throughout the process of their transformation. The biological transformation and/or composting process took place spontaneously in the middle of nature without human intervention.

3.2. Potential for Organic Nitrogen Mineralization

The curves in Figure 1 below show, respectively, the potential for organic nitrogen mineralization of each ROP in controlled conditions incubation tests in a laboratory.

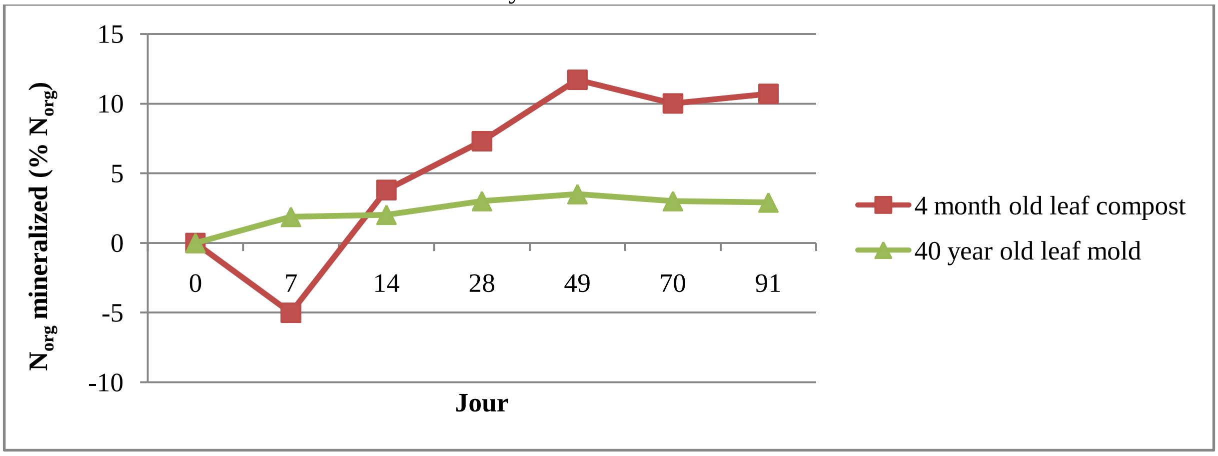

Fig. 1 Mineralization of organic nitrogen of the two ROPs

Notwithstanding, the reorganisation and/or immobilization phase (no mineralization) up to less than five percent (-5%) of organic nitrogen observed during the first week of incubation likely related to excessive mineralization of compost organic matter, the maximum 4-month-old organic compost nitrogen mineralization coefficient still reaches +11.7% at the end of the 91st incubation day, so it is a medium-term supply of a significant amount of nitrogen from the organic nitrogen form. This nitrogen immobilization is very common especially for green waste composts. The studies published in April 2002 by the Chambre d'Agriculture Bretagne (CRAB) report a major reorganization phase up to less than -11% on the 14th day but which ends with a percentage of organic nitrogen mineralized in the end by +1% or + 10% [46].
In addition, the mineralization of organic nitrogen from soils aged 40 years is very slow and low with only a maximum mineralization coefficient of 3.5% at the end of the 91st incubation days. These values are close to the values found in other work on the mineralization of organic nitrogen on some composts of green waste that have undergone a long maturation phase greater than 12 months [47,48]. We clearly see here that the organic nitrogen of the PROs with stable character as the case of these soils aged 40 years is slowly mineralizing. These leaf loams will therefore be preferentially used to maintain the organic carbon stock of the soil (organic amendment) and not as nitrogen fertilizer. Thus, Table 2 below summarises the nitrogen supply dynamics of each ROP.

Table -2 Summary of nitrogen fertilizer values for each ROP

ROPs	**Immediately available nitrogen ($N\text{-}NH_4^+ + N\text{-}NO_3^-$)**		**Organic nitrogen available in the medium term (mineralizable within one year of intake)**		**Organic nitrogen available in the long term (mineralizable in subsequent years)**	
	(% Ntotal)	kg/t ofRM	% Norg	kg/t ofRM	% Norg	kg/t ofRM
4 month old leaf compost	13.46	2.32	11.7	1.97	88.3	14.89
40-year-old leaf mold	0.38	0.07	3.5	0.65	96.5	18

The immediately available nitrogen is the mineral fraction of the product ($NH_4^+ + NO_3^-$). The available medium-term nitrogen is the fraction of the organic nitrogen that can be mineralised in the year according to the results obtained during incubation tests under controlled conditions in the laboratory. Finally, the nitrogen available in the long term represents the organic nitrogen that can be mineralized in subsequent years.

4. CONCLUSION

The nitrogen effect of the ROPs is therefore intimately linked to the composition of waste entering the manufacture of each ROP, the age of the ROP, the biological process of transformation and/or composting, etc. The older the ROP is (old), the slower the release of nitrogen (slow decomposition) due to the stability of the organic matter. The nitrogen effect is therefore lower for leaf compost aged 40 years compared to compost aged 4 months. However, the transition of nitrogen mineralization (laboratory) data to producer usable nitrogen efficiency (in the crop field) remains challenging, as many field phenomena could interfere (composition of the organic product, dose, mode and frequency of intake, soil type, climate, cultural succession, cultural practice, etc.).

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