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Study of the Agronomic and Ecological Potential of Leaf Compost Derived from Residues from Distillation of Essential Oils from Leaf Cloves: Case of Leaf Compost Aged Between 20 and 40 Years Collected at the Ambodimanga II Distillation Site, Rural Municipality of Maromitety, Vavatenina District, Analanjirofo Region (Madagascar).

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ABSTRACT: The Analanjirofo Region (Madagascar) is home to a large number of bush stills located throughout each glove zone used primarily for the distillation of clove leaf essence. The distillation of leaf species since its introduction in Madagascar in 1911 (Ramalanjaona and Jourdan, 1961; Dufournet, 1968) generates a large quantity of waste, specifically leaf waste, therefore a significant source of fertilizer material of waste origin (Fmwo). However, these residual leaves from distillation have remained untapped since that time. The studies we conducted on the sample of leaf loam aged about 20-40 years collected on the distillation site of Ambodimanga II, district of Vavatenina, Analanjirofo region (Madagascar) attest to the importance of the agronomic value of this type of fertilising material of waste origin (Fmwo). Indeed, among other things, these leaf earths have a very interesting amending value with an organic matter stability index (OMSI) more than 70% OM and a fairly satisfactory (average) fertilizer value. In short, the addition and/or application of these leaves to the soil helps to correct the deficiency of soil organic matter and is also an interesting source of nutrients for crops.

KEYWORDS : leaf loam, Fmwo, compost, organic amendment, bush still, fertilizer, safety

I. INTRODUCTION

Madagascar, especially the Analanjirofo Region is world famous for its production of nails and essences of cloves leaves. Moreover, Madagascar is the only country in the world that produces and exports clove leaf oil. Note that the clove (*Syzygium aromaticum*) is a tree introduced in Madagascar since the late nineteenth century. The clove is mainly cultivated for its nails, which are the flower buds picked before they bloom. Cloves are also grown to extract a very rich eugenol essence. Clove essence is extracted from different clove organs: nails, claws, leaves and antofles. (G. RAMALANJAONA and E. JOURDAN, 1961). Many studies have been carried out on plantings and extraction techniques of clove oil, but little or no study has been carried out on waste from distillation of leaf oil. Whereas in the past, this waste from the distillation of leaves can indeed serve as compost promoting the growth of taro or banana trees or other major agrarian speculations of the region (vanilla, clove, etc). They can also be used as a substrate for the rearing of a high-quality edible fungus, *Volvariavolvacea* (Bouriquet 1941, 1946), but this use seems lost today. The present study aims to examine through classical agronomic analyses (physico-chemical and biochemical analysis) the agronomic value of this leaf soil derived from distillation residues of clove species in order to assess their amending value (ability to provide stable humus to the soil) and their fertilising value (ability to provide major and secondary nutrients to the crop). In addition, in order to ensure the use of the Fmwo in agriculture, Innocuity tests were also carried out. These include the assays of the Elements of Metallic Traces (EMTs) as well as the Compounds of Organic Traces (COTs) that may be present in these soil of leaves.

II. MATERIALS AND METHODS

II-1-Potting soil

This is leaf loam aged approximately 20 to 40 years old collected at a distillation site of essential oils of clove leaves located on the Fokontany of Ambodimanga II, district of Vavatenina, Analanjirofo Region. Note that this leaf soil is the fruit of the natural decomposition without human intervention of the residues of leaves (stripped of essential oils)

resulting from the complete distillation of essential oil of leaves of cloves. Indeed, these residues of leaves once the distillation operation is completed; they will be voluntarily abandoned by the operators-distillers all around the site and/or shed of distillation without even the project of subsequent revalorization. Thanks to the pedoclimatic conditions of the Analanjifofo Region (hot and humid), these leaves residues decompose spontaneously and that over time, they will give rise to soils rich in stable, pre-humified or even humified organic matter (very stable).

II-2-Physico-chemical analyses

The physico-chemical analyses to be carried out as part of this experiment are : determination of the dry matter (DM), total organic matter (TOM), total organic carbon (TOC), total nitrogen, C/N ratio, pH, electrical conductivity (EC), nitric nitrogen (NO_3^-), ammoniacal nitrogen (NH_4^+), determinations of major fertilizing elements including phosphorus (P_2O_5), potassium (K_2O) and secondary elements CaO, MgO and Na_2O .

-Determination of dry matter (DM) is performed according to standard protocol (NF ISO 11465, 1994): drying 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,

-the total organic matter content (OM in % of DM) was determined by the mass loss when the sample was calcined at 550°C for 02 hours in a furnace (NF U 44-160 of November 1985 or European standard EN 15935,2009),

-Total organic carbon (TOC) will be determined by wet or sulfochromic oxidation (NF ISO 14235, 1998),

-total nitrogen by the Kjeldhal method (NTK) (EN 13342, October 2000),

-the pH measurement is carried out on aqueous suspensions according to AFNOR NF ISO 10390 of November 1994 and the pH measurement (0,1 pH unit) is carried out directly by reading on a combined electrode pH meter,

-specific electrical conductivity (EC) is measured on a 1/5 aqueous extract (compost mass/solution volume) at a temperature of $(20\pm 1)^\circ\text{C}$ (NF ISO 11265), -ammoniacal and nitric nitrogen by extraction with potassium chloride solution (KCl 1M) and then determined by colorimetric method. Absorbance is measured at 543nm for nitrates while ammonium absorbance is measured at 630 nm (Bremner 1965, Guiot 1975),-the C/N ratio calculated from TOC measured by sulfochromic oxidation or chemical oxidation and total nitrogen obtained by the Kjeldhal method (N-NTK),

-the determination of the phosphorus present in the compost sample consists in mineralizing the sample in the regal water (1 part HNO_3 for 3 parts HCl) followed by determination by visible spectrophotometry, that is to say colorimetric determination with ammonium molybdate called also Method Briggs (AFNOR NF EN 1189,1997),

-the determination of calcium (CaO), magnesium (MgO) and sodium (Na_2O) consists of mineralizing the sample in regal water (mixture of HCl and HNO_3 3 3/1) for 16 hours at room temperature, then boiling with reflux for 2 hours. The concentrations of each element are then determined using spectrometric method more precisely by high-frequency induced plasma atomic emission spectroscopy (ICP-AES), (ISO 22036,2008)

II-3- Biochemical Analysis

II-3-1- Biochemical Fractionation

This is the biochemical fractionation of organic matter in the soil sample in soluble fraction (SOL), equivalent fraction of hemicellulosic (HEM), equivalent fraction of celluloses (CEL), equivalent fraction of lignin (LIC) according to the Van Soest method described in FD U44-162 (AFNOR, 2016). This method consists of successively separating the 4 fractions of OM assimilated to biochemical fractions present in Organic residual Products (ORPs) (soluble fraction (SOL), hemicellulose (HEM), cellulose (CEL) and lignin (LIG)) using a fibre extractor, determined by weight difference after attack and then expressed in % of total OM or % of total organic C of organic matter. Soluble organic matter (referred to as SOL) is obtained by double hot water solubilization, followed by solubilization in a neutral detergent (NDF or Neutral Detergent Fiber) (sodium dodecyl sulfate). The organic fraction obtained after extraction with NDF minus that obtained after extraction with acid detergent (ADF or Acid Detergent Fiber) corresponds approximately to the amount of hemicellulose (HEM). The organic fraction obtained after extraction with ADF minus that obtained after extraction with (ADL or Acid Detergent Lignin) or commercial solution of 72% sulphuric acid which solubilizes the celluloses corresponds of course approximately to the cellulose content (CEL). Finally, the organic fraction obtained after extraction with ADL corresponds to the lignin content (LIC) contained in the sample. Thus, five samples of 1g dry and crushed at 1mm are placed in crucibles to which 1g of calcined sand is added.

Four extractions are performed successively:

-Extraction of the soluble fraction in hot water (SOL water) (extraction in 100ml of boiling water for 30minutes).

-Extraction of water soluble fraction + neutral detergent (SOL NDF) (extraction in 100ml of boiling water for 30 minutes then in 100ml of hot neutral detergent for one hour, NDF).

-Extraction of hemicellulosic fraction (HEM) (1hour in 100ml in hot acid detergent, ADF).
 -Extraction of the cellulosic fraction (CEL) (3 hours in concentrated sulphuric acid solution, ADL).

The residue is considered to be the lignin fraction (LIC).

After each extraction, the residual organic fraction was calculated based on the mass loss of the sample (the sample is dried at 105°C for at least 16 hours and then calcined at 480°C for 6 hours). The calculations of the different fractions are based on:

$$SOL_{eau} (\% OM) = 100 - MO_{water}$$

$$SOL_{NDF} (\% OM) = OM_{water} - OM_{NDF}$$

$$HEM (\% OM) = OM_{NDF} - OM_{ADF}$$

$$CEL (\% OM) = OM_{ADF} - OM_{ADL}$$

$$LIC (\% OM) = OM_{ADL}$$

With OM_{water} , OM_{NDF} , OM_{ADF} , OM_{ADL} , the proportions of OM measured in the residues after extraction

II-3-2-Carbon mineralization kinetics

Controlled Conditions Incubations (CCI) were conducted over a period of three (03) days to assess the carbon mineralization of carbon from the leaf soil in question once brought to the ground. These CCI were conducted in accordance with FD U44-163 (AFNOR, 2019).

Thus, 25 g of dry soil at 105°C, and a mass of compost such that the organic carbon input represents 0.2% of the dry soil mass (i.e. 2,000 mg of carbon/kg of dry soil) are incubated in the dark, at a temperature of 28°C for 03 days, The samples are moistened to 65% of the field capacity (8.12 g of water per 25 g of soil), optimal conditions for mineralization. The control (soil with no organic input) was also incubated. Each treatment is repeated 3 times. Mineralized C-CO₂ was collected after 1, 2, and 3 incubation days.

The mineralization of C is measured by trapping CO₂ in soda-ash (Freijer and Bouten 1991). Tubes containing 20 ml of NaOH 1M are placed in the incubation jars. Soda reacting with CO₂ precipitates in the presence of 5 ml of BaCl₂. The remaining soda is titrated with 0.1M HCL until turning a colored indicator (thymolphthaleine)

II-3-3-Organic Matter Stability Index

The OMSI (Organic Matter Stability Index) indicator is calculated on the basis of biochemical fractions and mineralized C in 3 days during incubations in a soil (Ct3) or:

$$OMSI (\% OM) = 44.5 + 0.5 SOL - 0.2 CEL + 0.7 LIC - 2.3 MinC3 (I)$$

With SOL, CEL and LIC the biochemical fractions (% OM) and MinC3, the proportion of organic C mineralized during the first 3 days of incubation.

The OMSI is a good estimator of the amending value (Lashermes, et al, 2009). This OMSI index is a rapid laboratory assessment of the coefficient K1 (Coefficient Iso-humique) of an organic product, a coefficient usually obtained from full-field experiments of medium to long duration (Lionel Jordan-Meille, Christian Morel, Xavier Salducci, Julien Michaud.2018)

II-4- Innocuity tests: determination of MTEs and OTCs

The safety of these leaf loams, which are the subject of this study, is assessed mainly by its content in metallic trace elements (MTEs), and its content in organic trace compounds (OTCs).

II-4-1- Determination of metallic trace elements (MTEs)

The determination of the possible presence of the MET consists first of extraction with water regale (1 HNO₃ unit for 3 HCl units) in accordance with ISO 11466,1995, and then to measure the elements extracted by this regal water by atomic emission spectrometry (ICP-AES) using the standardized method ISO 22036,2008. The relevant elements in this study are: arsenic (As), cadmium (Cd), chromium (Cr), cobalt (Co), copper (Cu), nickel (Ni), lead (Pb), zinc (Zn).

II-4-2- Determinations of organic trace compounds (CTOs)

CTOs subject to strict verification and control in the compost sample are: (i) monocyclic aromatic hydrocarbons (M.A.Hs) such as Benzene, Toluene, Ethylbenzene, o-Xylene, m-Xylene, p-Xylene, Styrene; (ii) and some Polycyclic Aromatic Hydrocarbons (PAHs) such as Fluoranthene, Benzo (b) Fluoranthene, Benzo (a) Pyrene and the total of 7 congener PCBs classified as Persistent Organic Pollutants (POPs).

-Monocyclic aromatic hydrocarbons (Benzene, Toluene, Ethylbenzene, o-Xylene, m-Xylene, p-Xylene, Styrene) are determined by gas chromatography coupled to mass spectrometry (GC/MS) in accordance with international standard ISO 22155, 2011;

-polycyclic aromatic hydrocarbons (Fluoranthene, Benzo (b) fluoranthene, Benzo (a) pyrene) are determined by high-performance liquid chromatography (HPLC) method (ISO Standard 13877,1998), -7PCBs congener in occurrences PCB n° 28, 52, 101, 138, 153 and 180 are determined by gas chromatography and electron capture detection (ISO 10382, 2002; AFNOR XPX33-012, 2000)

III. RESULTS AND DISCUSSIONS

III-1-Physico-chemical analysis

The following table summarizes the results of physico-chemical analysis carried out on Ambodimanga II soil samples.

Table 1: Main physico-chemical characteristics of Ambodimanga II leaf mold

Analysis	Units	On crude	On dry
Dry matter	%	61,74	
pH water	Unit pH	6,1	
Electrical conductivity	$\mu\text{S}\cdot\text{cm}^{-1}$ à 20°C	691	
Organic Matter (OM)	%	46,21	74,84
Total Organic Carbon (TOC)	%	26,30	42,62
Kjeldahl nitrogen	%	1,87	3,02
Ratio C/N	Without unit		14,11
Nitric nitrogen	mg N/Kg	69	112
Ammoniacal nitrogen	mg N/Kg	<10	<16
Phosphorus (P_2O_5)	%	0,296	0,479
Potassium (K_2O)	%	0,139	0,225
Calcium (CaO)	%	1,455	2,356
Magnésium (MgO)	%	0,437	0,708
Sodium (Na_2O)	%	0,09	0,145

The leaf mold of Ambodimanga II is very rich in stable organic matter on the other hand means or even qualified poor in major fertilizing elements. Indeed, because of the advanced age of this leaf loam, the mineral elements are probably exposed to phenomena of excessive leaching especially by rainwater. For this reason, the electrical conductivity value of this leaf soil is extremely low. Similarly, the sodium (Na_2O) and nitrates (NO_3^-) levels are extremely low, probably because of these leaching phenomena and/or by the phenomenon of deep drainage, particularly by rainwater. However, it is possible that during the formation of humic substances, significant amounts of salts could potentially fix on the stabilized MO (Francou, 2003) and thus cause the decrease of electrical conductivity (EC).

The pH here is slightly acidic 6.1. This reflects anaerobic conditions during the biological transformation process of these wastes, leading to excessive acidity of the soil and/or organic amendment obtained. It should be noted that the leaf mold in question has not undergone any reversal throughout the process of their biological transformation. The biological process of their transformation into soil was spontaneously carried out in the middle of nature without human intervention



III-2- Biochemical Analysis

III-2-1-Biochemical fractionation

Table 2 provides the percentages of lignin, cellulose, hemicellulose and soluble substances in the Ambodimanga II leaf mold sample, expressed as a percentage of OM.

Table 2 : Biochemical fractionation results expressed as a percentage of OM

Biochemical fractionation of OM	Result in % OM
Organic compounds insoluble in neutral detergent NDF (% of OM)	80
Organic compounds insoluble in acid detergent or ADF (% of OM)	70
Sulphuric lignin ADL ADL (% of OM)	42
Soluble organic compounds "SOL"	20
Hemicellulose "HEM"	10
Cellulose "CEL"	28
Lignin and cutine "LIC"	42

Neutral Detergent Fibre (NDF); Acid Detergent Fibre (ADF); Acid Detergent Lignin (ADL); with Cellulose: CELL = ADF – ADL respectively; Hemicellulose: HEMI = NDF – ADF; Lignin: LIGN = ADL; Soluble Fraction: SOLU = 100 – NDF

III-2-2-Carbon mineralization kinetics

Figure 1 shows the apparent mineralization kinetics of the organic carbon in our soil sample tested over the three (3) incubation days, calculated by the difference between the CO₂ release from the treatment and that from the soil alone

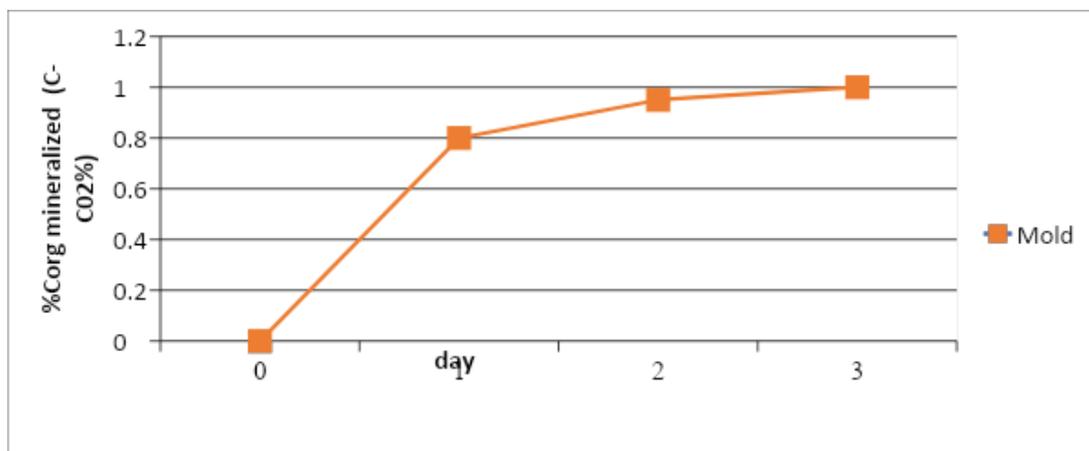


Figure 1 : Cumulative Carbon Mineralization Kinetics as % of Initial C

Only about 1% of TOC was mineralized after 3 days of incubation. The rate of mineralization is therefore very slow due to the very high stability of the organic matter of the leaf mold sample examined.

III-2-3- Organic Matter Stability Index (OMSI)

The OMSI is calculated using the formula (1) cited above. Thus, the OMSI value of the Ambodimanga II mold sample is:

OMSI as % of organic matter 76%

OMSI kg/T dry product 568.78

OMSI kg/T of gross product 351.20

Indeed, these leaf loams have an excellent amending value with more than 76% of the organic matter therefore endowed with a considerable humogenic potential and qualified as « excellent humogenic potential». This result corroborates the results found by Sabine Houot (2012) on green waste composts



III-3- Results of Innocuous Tests

The results of Innocuous Criteria Reviews are shown in the following Table 3:

Table 3: Safety Criteria Analysis Results

Elements to be monitored	Concentration (mg/kg DM)	Concentration (mg/kg CM)	Acceptable Limit Values (mg/kg DM) SAQ-TERROM Reference Acceptable	Limit Values (mg/Kg DM) NFU 44-051
ETMs				
Arsenic (As)	0,1	0,06	≤16	18
Cadmium (Cd)	0,14	0,08	≤ 2,7	3
Copper Cu)	113	69,76	≤ 270	300
Nickel (Ni)	8	4,93	≤ 54	60
Lead (Pb)	7	4,32	≤162	180
Zinc (Zn)	52	32,10	≤540	600
Chrome (Cr)	15	9,26	≤108	120
CTOs				
H.A.MS (benzene, toluene, ethylbenzene, o-xylene, m-xylene, p-xylene, styrene)	<0,11	<0,067	≤5	
fluoranthene	0,05	0,030	≤3,6	4
Benzo (b) fluoranthene	< 0,01	0,0061	≤ 2,3	2,5
Benzo (a) pyrene	< 0,01	0,0061	≤ 1,4	1,5
Total H.A.Ps	0,10	0,061	≤3	
P.C.B.'s Total (n° 28, 52, 101, 118, 138, 153,180)	<0,011	<0,0067	-	0,8

In general, the elements examined are all well below the tolerable limit values, thus complying with the thresholds defined in the NFU 44-051 standards on organic amendment and the SAQ-TERROM or even other equivalent standards required on compost qualities (Ecolabel, etc.). The agricultural use of Ambodimanga II leaf loam does not therefore cause major environmental risks.

IV. CONCLUSION

This study highlighted the agronomic potential of leaf soil samples from distillation residues of clove leaf species. Notwithstanding, the age of soil sampled, it can be concluded that their uses in agriculture are very promising. Indeed, we have just found that these leaf loams are qualified as excellent «organic amendment» compliant even with the French Standard NFU 44-051 on organic amendments. With its stability index in organic matter OMSI greater than 70% OM, they can be qualified as an organic product (ORP) with high humogenic potential. In addition, these leaf loam also contain significant nutrients. The greater the amount of soil input, the greater the amount of fertilizer that is produced. At the same time, the examination of the criteria of Innocuous shows the ecological interest of these leaf loam because they are free of all the undesirable elements for the environment (MTEs and CTOs).

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