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# Comparative Study of the Amending Value of the Two Organic Residual Products (ORPs) Derived from Distillation Activities of Clove Leaf Species : case of 4-month-old leaf compost and leaf soil aged about 40 years.

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**ABSTRACT:** Since the introduction in Madagascar of the first distillation still of essential oil from clove leaves in 1911 (Ramalanjaona and Jourdan, 1961; Dufournet, 1968) little has been done on distillation waste in the form of leaves. However, in the past, this waste from distillation could indeed be used as compost to promote the growth of taro or banana trees or other major agrarian speculations in the country. They can also be used as a substrate for the rearing of a high-quality edible fungus, *Volvaria volvacea* (Bouriquet 1941, 1946), but this use seems lost today. The study we carried out on two original distillation ORPs shows very promising results. The ORPs resulting from distillation activities have indeed very interesting indices of organic matter stability (OMSI) with respectively nearly 58% OM for leaf compost aged 4 months and 76% OM for those aged around 40 years. They can therefore be described as excellent organic amendments with a high humige potential, that is to say that once incorporated into agricultural soils, these two ORPs will actively contribute to the maintenance of the humic stock of soils.

**KEYWORDS :** Compost, leaf soil, biochemical fractionation, amending value, fertilizing value, mineralization, humogenic potential

## I. INTRODUCTION

The amending value of ORPs is defined as their ability to maintain and/or replenish soil organic matter contents. The interest of an organic amendment is related to its potential to transform into humus, depending on the origin of the raw materials and its elaboration (composting process). The assessment of an organic product in terms of amendment is of paramount importance. The amending value of ORPs can be assessed by measuring the mineralization of their organic carbon during incubation in a mixture in soil, under controlled laboratory conditions, according to a standardized protocol (FD U44-163). The Organic Matter Stability Index (OMSI) is an indicator of the amount of organic matter likely to remain in the soil as humic matter over the long term, and therefore an indicator of the amending value of ORP (Lashermes and al., 2009; Peltre and al, 2012). It is calculated based on the results of a biochemical fractionation of the ORPs (FD U44-162) and the mineralized carbon content after three (3) incubation days (FD U44-163, June 2019). The higher the OMSI, the better the product is to maintain this stock over the long term. From this index the coefficient K1 (Coefficient Iso-humic) of an organic product can be assessed in the laboratory, a coefficient usually obtained from full-field experiments of medium to long duration (Lionel Jordan-Meille, Christian Morel, Xavier Salducci, Julien Michaud, 2018). For example, in order to assess the contribution effect of a ORP on the properties and the level of organic matter in the soil, it is not uncommon to have to wait 4 to 5 years (ADEME, 2016), or sometimes 15 or 50 years sometimes (Clément Peltre & Sabine Houot, 2011; Hien, 2004). In addition, several factors will have to be taken into account, in particular the different pedoclimatic conditions, etc.

The ultimate objective of this study is to estimate the amending value of the two original ORPs of distillation activity of essential oils from clove leaf through the results of studies and incubation tests under controlled conditions (organic carbon mineralization C) and OMSI index analysis results. Note that the latter is a way to estimate in a more efficient and reliable way the amending value of the ORPs.

## II. MATERIALS AND METHODS

### II-1-Organic waste product

The two ORPs, the objects of this study are:

- Compost based on waste bush alembic leaf aged four 4 months provided by the company Iazafo Compost. This compost was made from leaf residues from the complete distillation of essential oils from the leaves of the cloves,
- About 40 years old leaf loam collected at the Ambodimanga II distillation site, Maromitety Rural municipality, Vavatenina District, Analanjrofo Region (Madagascar).

These leaf loams came from of the natural decomposition without human intervention of leaf residues (stripped of essential oils) abandoned voluntarily (without a subsequent recovery project) by distillers around the distillation site after each leaf distillation operation.

These distillations were carried out since 1980 and even before for some sites. Thanks to the pedoclimatic conditions of the Analanjrofo 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).

Here it is necessary to specify that this is the result of survey and interview with notables (*tangalamena*) and local residents, and especially the owners of distillation stills at the Ambodimanga II site, which gave us valuable information on the age of these soils formed around the Ambodimanga II site (see photo n° 1 below). According to them, this distillation site has been operational since 1979, an estimated 40 years of existence in 2020.



Photo n°1: Waste leaf swath from the Ambodimanga II site (personal source)



Photo n°2 : Sample collection of Ambodimanga II leaf loam (personal source)



Photo n°3 : Bundle of fresh clove leaves before distillation on bush still (personal source)



Photo n°4 : Fagots de feuilles de giroflier transporter à dos d'hommes avant distillation (personal source)

## II-2- Biochemical Fractionation

This operation consists of fractionating the organic matter of a ORP into four biochemical families on the basis of their successive solubility in extraction reagents: soluble substances (SOL-rated), hemicellulose (HEM-rated), cellulose (CEL-rated) and lignin and cutine (noted as LIC). This fractionation takes advantage of the differences in properties of biochemical compounds to ensure their extraction. Hemicelluloses are soluble in weak acidic solutions, cellulose is hydrolyzed in a strong acidic medium and lignin is insoluble even in a strongly acidic medium. Many fractionation methods are proposed in the literature, some of which are intended for the determination of a single constituent (Morel and al., 1986; Garcia and al., 1992). Other methods are based on sequential extractions. The objective is to determine the content of the different families of biochemical constituents of the ORP, the main ones being in general, water-soluble compounds, hemicellulose, cellulose and lignin. The most cited fractions in the literature are van Soest fractionation: (van Soest & Wine, 1967) and fractionation according to ASTM E1788-95<sup>e1</sup> (1995) cited by Kelly (2002). They are based on hydrolyses of biochemical constituents. Currently, there is a standard or more specifically a FD U44-162 documentation booklet (AFNOR, 2016) replacing the experimental standard XPU 44-162 (AFNOR, 2016) to characterize organic matter by biochemical fractionation on organic amendments. This FD U 44-162 standard allows the sequentially determination of constituents by treatment with neutral detergents (NDF) and acid (ADF) and sulphuric acid (ADL).

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 ) such as 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 quantity of hemicellulose (HEM), the organic fraction obtained after extraction with ADF minus that obtained after extraction with ADL (Acid Detergent Lignin or commercial solution of 72% sulphuric acid which solubilizes the celluloses) corresponds approximately to the cellulose content (CEL). Finally, the organic fraction obtained after extraction at ADL corresponds to the lignin content (LIC) contained in the sample.

The content of cellulose, hemicellulose and lignin in the samples can be determined by successive subtractions. In addition, it is difficult for us to obtain the necessary chemical reactive differences under this experimental protocol FD U 44-162, including acid detergents and neutral detergent. To overcome this disadvantage, we developed a biochemical fractionation protocol based on bibliographic research. This protocol is described in Figure 1. This protocol differs from the experimental protocol FD U44-162 (AFNOR, 2016) in that the steps of solubilization to neutral and acid detergents are replaced by solubilizations to hot water and sulphuric acid (5N) and that, on the other hand, each

solubilization step is followed by a centrifugation step in order to allow a better settling and thus avoid material losses. Thus, the soluble organic fraction, SOL is determined by contact of the shredded waste at 2 mm with water at 90°C for one hour. The liquid/solid ratio is 50. The solid residue is dried and used for hemicellulose extraction. Extraction of hemicellulose is carried out hot (90°C) for 1 hour in an acidic medium with (H<sub>2</sub>SO<sub>4</sub> 5 N) with a liquid/solid ratio of 40. The suspension is then diluted with distilled water. The solid residue is dried and used for the cellulose extraction step. Determination of cellulose is performed according to standard procedure ASTM E1788-95<sup>e1</sup> (1995), which consists of acid hydrolysis (H<sub>2</sub>SO<sub>4</sub> 72%), at 35°C for 1 hour, followed by dilution and autoclaving at 121°C and at 15 PSI for 1 hour.

SOL, HEM and CEL are quantified by measuring TOC on liquid as a direct determination using the wet method such as sulfochromic oxidation in accordance with NF ISO:14235, 1998. The non-extractable residue NER, consisting of both woody compounds and plastics, is determined by measurement of solid TOC and by calcination either on suspended solid sample in a liquid medium by sulfochromic oxidation as described in NF ISO: 14235, 1998. It should be noted here that the composts studied do not contain inerts (in particular no plastics) and it can therefore be assumed that the non-extractable fraction NER does indeed correspond to woody compounds plus some cutines, (Djakovitch, 1988)

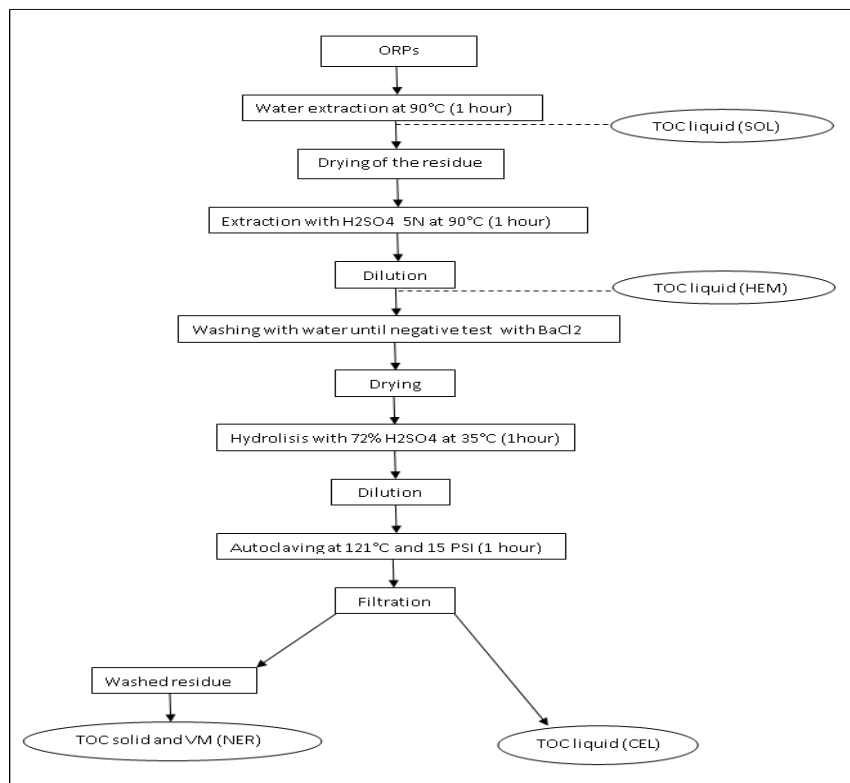


Figure 1: Biochemical fractionation in SOL, HEM, CEL and RNE (source: Faouzia, 2008)

**II-3- Incubation test : carbon mineralization kinetics**

Even if the results of incubation studies during the first three days are sufficient for the calculation of OMSI of each ORP later, we have nevertheless continued these studies of Incubations in Controlled Conditions (ICC) up to 91 days to assess the carbon mineralization of each ORP once it is brought to the ground. Indeed, this incubation time of 91 days, allows us to simulate the effect on the field of culture of each ORP over 12 months following the intake. These ICC studies 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 91 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. The mineralization of C is measured by trapping CO<sub>2</sub> in soda (Freijer and Bouten, 1991). Tubes containing 20 ml of NaOH



1M are placed in the incubation jars. Soda reacting with CO<sub>2</sub> precipitates in the presence of 5 ml of BaCl<sub>2</sub>. The remaining soda is titrated with 0.1M HCL until turning a coloured indicator (phenolphthalein) The measurement points are made at times of: 0 day, 1 day, 3 days, 7 days, 14 days, 21 days, 28 days, 49 days, 70 days and 91 days. The mineralization of C is expressed as a difference to the mineralization of a soil that has not received input (control and/or reference) and as a percentage of the amount of carbon added. Expression of results:

$$C_{comp} = (C_{soil+comp} - C_{soil\ only}) \frac{m_{soil}}{m_{comp}}$$

With

C<sub>comp</sub>: C of mineralized compost (g.kg<sup>-1</sup> dry compost)

C<sub>soil only</sub>: C-CO<sub>2</sub> mineralized for soil only (mg.kg<sup>-1</sup> dry soil)

m<sub>soil</sub>: dry mass of soil (g)

m<sub>scompost</sub>: dry compost mass (g)

C<sub>soil+ comp</sub>: Sum of C of compost and mineralized incubation soil (g.kg<sup>-1</sup> dry compost) Be expressed as a percentage of Corg according to the following formula :

$$C_{comp}(\%COT) = 100 \times \frac{C_{comp}(g.kg^{-1}dry\ compost)}{COT(g.kg^{-1}dry\ compost)}$$

Thus, the agronomic interpretation of each ORP according to their mineralization kinetics is given in the table below:

Table 1: Agronomic Interpretation (Celesta lab scale of values, 2012):

0 < Ct <sub>91</sub> < 15 %	15 < Ct <sub>91</sub> < 30 %	30 < Ct <sub>91</sub> < 50 %	Ct <sub>91</sub> > 50 %
-Strong stability -High Humic Potential -very low stimulation of microbial activity <u>Effect on the ground:</u> - Erosion control; -Improvement of the CEC and UK = (useful reserve)	-Good stability -Good humic potential -Low stimulation of microbial and earthworm activity <u>Effect on the ground:</u> - mainly structuring	-Intermediate stability -Medium Humic Potential -Average effect on microbial and earthworm biomass <u>Positive effect on structural stability</u>	-product not stabilized, -low humic potential, -stimulation of microbial activity. <u>Little long-term structuring effect.</u>

(Source : Salducci X., 2012)

#### II-4- 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 incubation in soil (Ct3) using the following equation (Lashermes et al, 2009; AFNOR, 2009):

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

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 represents a good estimator of the amending value of the ORPs. The biochemical fractions of organic matter determined according to the above principle are used to calculate the relative amount of organic matter in the product that is more resistant to mineralization and residual approximately one year after intake. This calculation results from a statistical correlation established between organic fractions and the percentage of mineralizable organic carbon after 3 days of incubation on the one hand and the residual carbon levels determined by extrapolation of mineralization kinetics carried out in controlled condition on the other hand (Lashermes, et al, 2009).

The OMSI can be expressed in % of organic matter (%OM) or kg/t of dry product (quantity of organic matter stable on dry) or in kg/t of gross product (quantity of organic matter stable on crude)

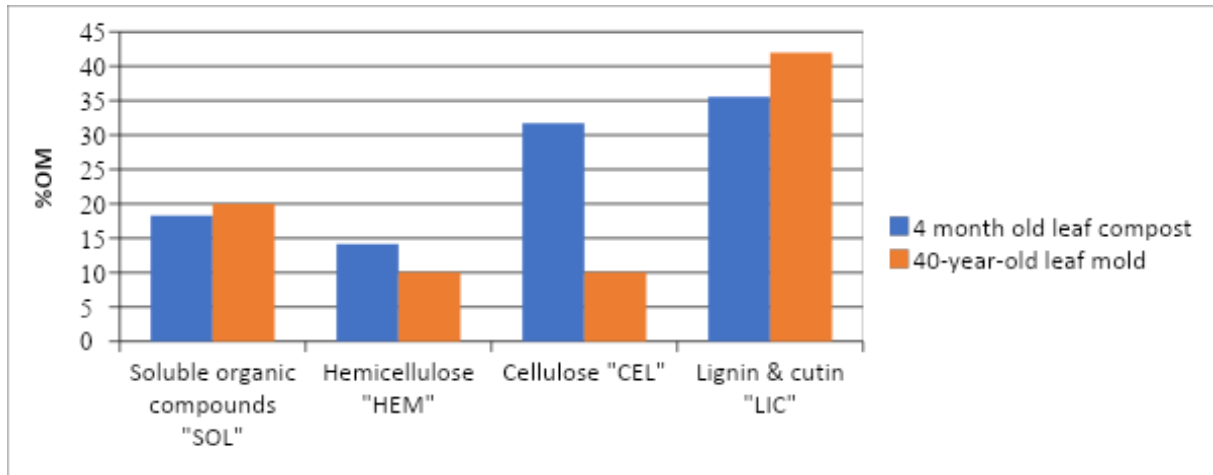


Figure 2: Biochemical fractionation of the two ORPs

In general, the organic matter of stills waste is rather high in fibre (cellulose and lignin) and relatively low in labile substance (sugar, etc.). But a priori, leaf soil seems to be richer in fraction recalcitrant to biodegradation (lignin). With age, organic matter probably transforms into a more stable humic substance.

### III. ORGANIC CARBON MINERALIZATION KINETICS

The curves in Figure 3 below show the kinetics of cumulative mineralization of carbon expressed in initial % C of ORPs

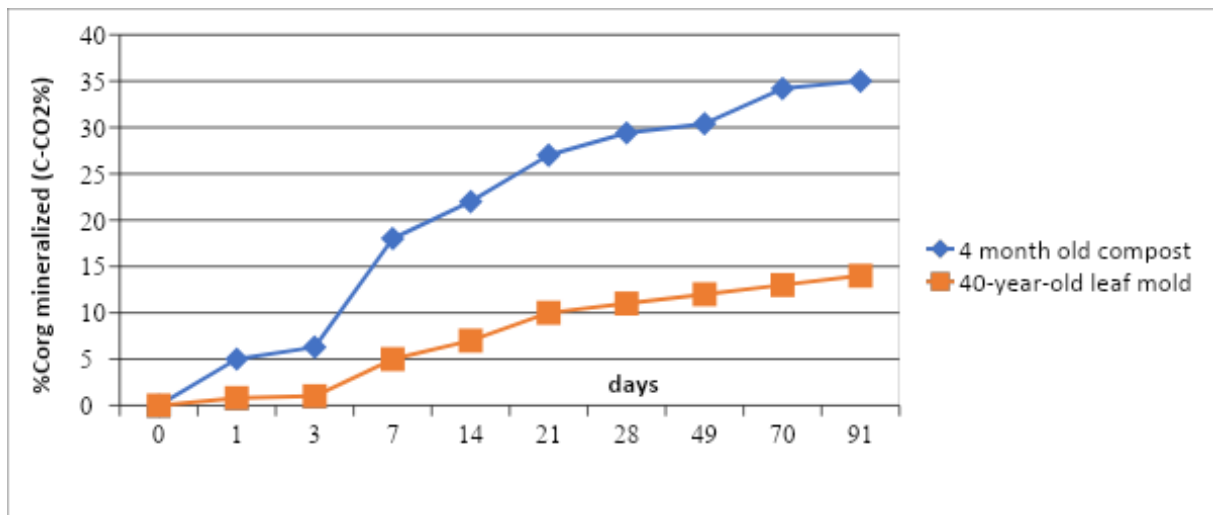


Figure 3 : Mineralization kinetics of the two ORPs

At the end of incubation, the mineralized carbon represents 35% of the carbon brought for the 4 months old compost. On the other hand, mineralized carbon accounts for only 14% of carbon input for leaf loam. Referring to the stability value scale of the ORPs mentioned in Table 1 above, leaf compost can therefore be described as “high stability” and “intermediate stability” for 04-month old leaf compost. The use in agriculture of compost aged 4 months will therefore significantly stimulate the activity of soil microorganisms. As for the use of 40-year-old leaf loam, because of their proven high stability, they will actively participate in their turn in the restitution of OM or even in the recovery of OM stocks from the ground. This glaring difference in carbon mineralization kinetics between these two ORPs of the same family is mainly related to age. Indeed, over time, 40-year-old leaf loam has been able to develop humic substances rich in functional groups (carboxyls, carbonyls, aromatic groups, etc.) that would allow them to



acquire a high stability of organic matter. The prolonged maturation phase plays a significant role in the stabilization and humification of OM (AUREA Agro Reporter, 2018).

### III-1- Organic Matter Stability Index (OMSI)

The OMSI is calculated using the formula (I) mentioned above. The OMSI value of each ORP is therefore graphically represented in figure 4 below :

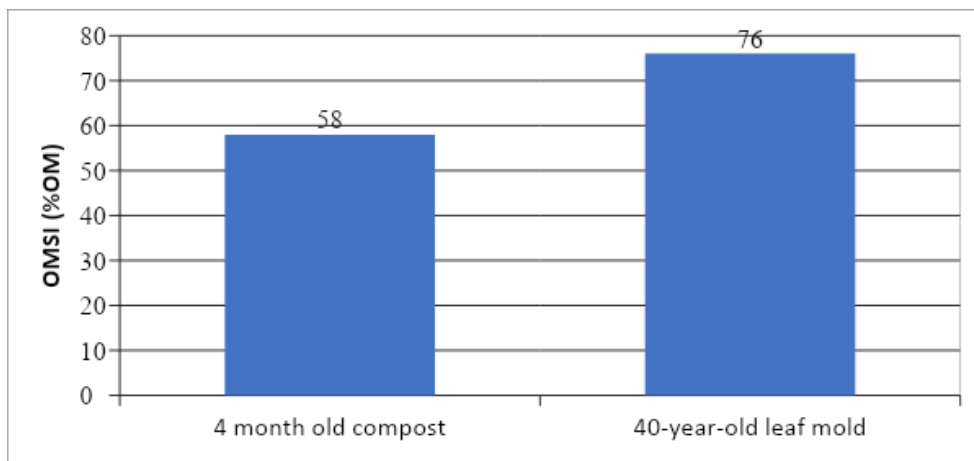


Figure 4 : OMSI index value of two ORPs

Once again, the high stability of organic matter in 40-year-old leaf loam has been demonstrated. According to the OMSI index results mentioned in the figure above, leaf compost has a much higher OMSI value than leaf compost that is only 4 months old with 76% OM and 58% OM respectively. So here, the 40-year-old leaf compost has more stability than the 4-month-old leaf waste compost. The older the ORP, the more important the OMSI. At the same time, the results of OMSI are significantly correlated with the stability estimation of organic materials obtained in the laboratory. In fact, leaf compost with a maximum mineralization coefficient of 14% TOC is classified here as more stable than that of compost of waste leaves aged 4 months whose maximum mineralization coefficient of organic carbon after 91<sup>st</sup> days is equal to 35% of TOC brought in. So the leaf loam has a very interesting amending value (humic yield) (OMSI =76% OM) compared to the leaf compost only 4 months old. However, this OMSI indicator is not used to discriminate between organic amendments (implying that one high OMSI product would be “better” than another low OMSI product). The OMSI is therefore an indicator of the stability of organic matter brought to the soil, which does not provide information on all the positive effects of organic matter on soil properties.

Structural stability, which conditions soil aeration and rooting, is increased by the addition of labile (easily degradable) organic matter rather than stabilized organic matter, as these are soil micro-organisms (by consuming these labile organic materials) which form the aggregates responsible for this stability. Low organic OMSI amendments therefore increase structural stability faster than high organic OMSI amendments. For other soil properties, such as water retention capacity or cationic exchange capacity (CEC), higher organic OMSI amendments will be most effective (Aurélié REIBEL & GERES, 2018).

### IV. CONCLUSION

The origin ORPs of distillation activities of clove leaf essences are all qualified as excellent organic amendments. Their main purpose is to restore the annual humic loss of agricultural plots (humus deficiency) and reactivate the “dormant” (or reduced) microbial biomass to maintain good soil functioning (AUREA Agro Reporter, 2018). However, the study we conducted clearly established the difference in amending power of each ORP when they come from the same basic substrates (waste leaves of bush stills). The old leaf soil shows a more interesting amending effect than composting less aged leaf waste (4 months). On the other hand, the latter, with its relatively small amending power, once incorporated into the soil further promotes the activity of soil micro-organisms and significantly improves or even strengthens the structural stability of the soil. The ideal will be to mix the two ORPs in order to optimize their agronomic effect.

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