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Physico-Chemical Characterization of Wood Ash for Agronomic Purposes

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Abstract

The oil refinery of the food products company SOPAL, located in the port area of TOGO, produces an average of 200 kg of eucalyptus wood ash per day. This ash is stored in large quantities within the premises of the refinery, without any disposal solution. The aim of this study is to identify the physico-chemical properties of SOPAL's wood ash for agronomic valorization. Physico-chemical analyses reveal the presence of major and secondary elements such as potassium (15.71 g/kg DM), phosphorus (27.90 g/kg DM), calcium (9.76 g/kg DM), magnesium (0.03 g/kg DM), sodium (1.24 g/kg DM), iron (1.49 g/kg DM) and manganese (7.82 g/kg DM). The pH is alkaline (12.97) with fairly high conductivity (24.10 mS/Cm). Heavy metals identified are cobalt (0.19 mg/kg DM), cadmium (0.10 mg/kg DM), lead (1.14 mg/kg DM), copper (19.77 mg/kg DM) and nickel (12.82 mg/kg DM). The results show that the ash contains considerable amount of major and secondary elements for agronomic uses.

Keywords

Industry, Wood, Ash, Heavy Metals, Valorization

1. Introduction

SOPAL is located in the free-port zone of Lomé. Alongside food products manufacture, the factory is specialized in the processing of crude oil into edible oil. Its combustion unit (furnaces) uses biomass fuel (eucalyptus wood), and generates an average of 200 kg of ash per day. To date, there is no means of disposal or recycling of the ashes. They are simply stored and buried on the refinery site. This situation is getting out of hand as space for stocking and burying the ashes in the refinery keeps diminishing.

Wood ash is most often used in agriculture for liming and potash fertilization [1] [2] [3]. It is sometimes combined with other substances to enhance its fertilising effects [4] [5] [6].

In West Africa, ash is used to enrich fallow land by proving minerals needed by plants [7]. The neutralizing or liming power of wood ash is due to its high concentrations of alkaline elements (calcium, magnesium, potassium), which raise the pH of the ash, mostly between 9.0 and 13.5 [8].

Wood ash (biomass ash) also contains microelements. Among these, elements such as cadmium (Cd) and lead (Pb) have no physiological function and are rather toxic to the soil, crops and humans [9]. Other microelements (Fe, Mn, Cu, Mo) are essential for plant growth, but in low concentrations. Thus, over a long term, repeated ash application can lead to accumulation of some microelements to toxic levels [10].

The composition of wood ash (biomass ash) is highly variable [11] [12]. It depends on a number of factors, including the species of wood and the part of the plant used, the type of soil and climate in which the trees were grown, as well as their storage and combustion conditions.

The aim of this study is to identify the physico-chemical properties of SOPAL wood ash in order to appraise the possible agronomic applications.

2. Material and Methods

2.1. Origin and Storage Place of the Wood Ash

Once recovered from the refinery, the ash is transported to the platform where poultry by-products are recycled into agronomic products at the agronomic laboratory of the University of Lomé, where it is stored in a bin (**Image 1**). The GPS coordinates of this work site are: latitude 6.174244 and longitude 1.210057.

2.2. Preliminary Ash Treatments

The stored ash (**Image 1**) is a mixture of daily samples taken over a period of one month. A quantity of 2.0 kg of wood ash from the "raw ash" bin was sieved with a 105-micron sieve (**Image 2**), in order to get an idea of the macroscopic composition of the ash and to facilitate its analysis in the laboratory. The experiment was carried out several times (10 times), in order to obtain an estimate of the quantity of coarse elements contained in the ash. The part of the raw ash that passed through the sieve (65% on average) is called "sieved ash" (**Image 3**) while the part that could not (35% on average) is called "residual ash" (**Image 4**).

2.3. Wood

A sample of the wood (**Image 5**) is recovered at the SOPAL's oil refinery, then ground into sawdust (**Image 6**) to be analysed at the laboratory.

2.4. Observations and Preliminary Tests Carried Out on the Ash

The various observations and tests carried out on the ash reveal its macroscopic composition.

In the raw ash, large fragments of magnetizable metal objects (**Image 7** and **Image 8**), charcoal (**Image 9**) and clinker (**Image 10**) were observed. After

Image 1. Wood ash from SOPAL oil refinery.

Image 2. 105 micron sieve.

Image 3. Sieved ash.

Image 4. Residual ash.

Image 5. Eucalyptus wood.

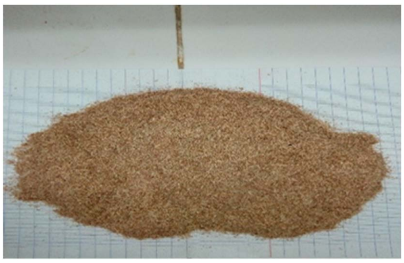

Image 6. Eucalyptus wood sawdust.

Image 7. Piece of metal.

Image 8. Metal scrap.

Image 9. Charcoal.

Image 10. Clinker.

sieving the raw ash, much smaller fragments of charcoal, clinker and scrap metals were found in the residual ash (Image 4).

2.5. Determination of the Physico-Chemical Parameters of the Sieved Ash

2.5.1. Measurement of PH and Electrical Conductivity (EC)

A 20 g mass of sample was dissolved in 100 ml of distilled water, then homogenized by shaking for 30 minutes and left still for 2 hours [13] [14]. Electrical Conductivity (EC) was measured at a ratio of 1:5 (sample/distilled water) [15] [16] [17]. pH and EC measurements were made directly on a SANXIN MP522 pH meter (Image 11).

2.5.2. Determination of Moisture Content (H_2O)

A sample was steamed at 105˚C for 24 hours to determine the moisture content [18].

2.5.3. Organic Matter (OM)

Organic matter content was obtained by loss on heating at 550˚C for 4 hours [14] [17].

2.5.4. Total Organic Carbon (TOC)

TOC in the samples was determined using the Anne Method (NF ISO 14235), based on sulfochromic oxidation in which chromium VI is reduced by organic matter to chromium III [19].

2.5.5. Total Phosphorus (TP)

Total phosphorus was determined in two stages [20]. The first step is digestion in an acid medium, which converts all the phosphorus present into orthophosphate. In the second stage, orthophosphate ions are determined and reacted with the molybdate and the antimony ions to form a phosphomolybdate complex. The latter is reduced with ascorbic acid in an acid medium to produce a blue coloration, the absorbance of which at 660 nm is proportional to the concentration of the orthophosphate ion. Phosphates were determined using a colorimetric analyser (Image 12). The color produced during reduction of the complex compound formed in the presence of orthophosphates or molybdate was measured at 660 nm.

2.5.6. Total Nitrogen (TN)

The Kjeldahl Method [21] was used for the determination of the total nitrogen. The organic matter in the sample was mineralized with hot concentrated sulfuric acid in the presence of a selenium catalyst. The distiller used was VELP SCIENTIFICA (Image 13). Total nitrogen content was determined using the following formula

$$C_N = 2C \times M_N \times \left(\frac{V_1 - V_0}{V}\right) \times 1000. \quad (1)$$

Image 11. pH-mèter/conductimeter.

Image 12. Spectrophotometer (SHIMADZU).

Image 13. Distiller of Nitrogen (VELP SCIENTIFICA).

With C_N nitrogen concentration, volume V_1 of sulfuric acid in millilitres (ml) required for dosing, sulfuric acid concentration C (in moles/litre), M_N molar mass of nitrogen, the volume V_0 of sulfuric acid in millilitres used for blank dosing, the volume V in millilitres (ml) of the test sample.

2.5.7. Metallic Trace Elements (MTE)

1) Mineralization and solubilization of samples

The solubilization method used was mineralization with aqua regia (1/3HNO_3 + 2/3HCl) in accordance with AFNOR NF ISO 11 460 of June 1995. It was carried out in a closed environment and at high temperature (110˚C - 150˚C).

2) Atomic Absorption Spectrophotometer (AAS) determination of chemical elements (Fe, Ca, Mg, Na, K, Mn, Cd, Pb, Cu, Ni and Co)

Determination of the chemical element content of the samples (Ca, Mg, K, Na, Fe, Mn, Cd, Cu, Pb, Co, Ni) was carried out in relation to the calibration curve (**Figure 1**) using a series of standard solutions of known concentrations. The standard solutions are therefore used to calibrate the measuring instrument.

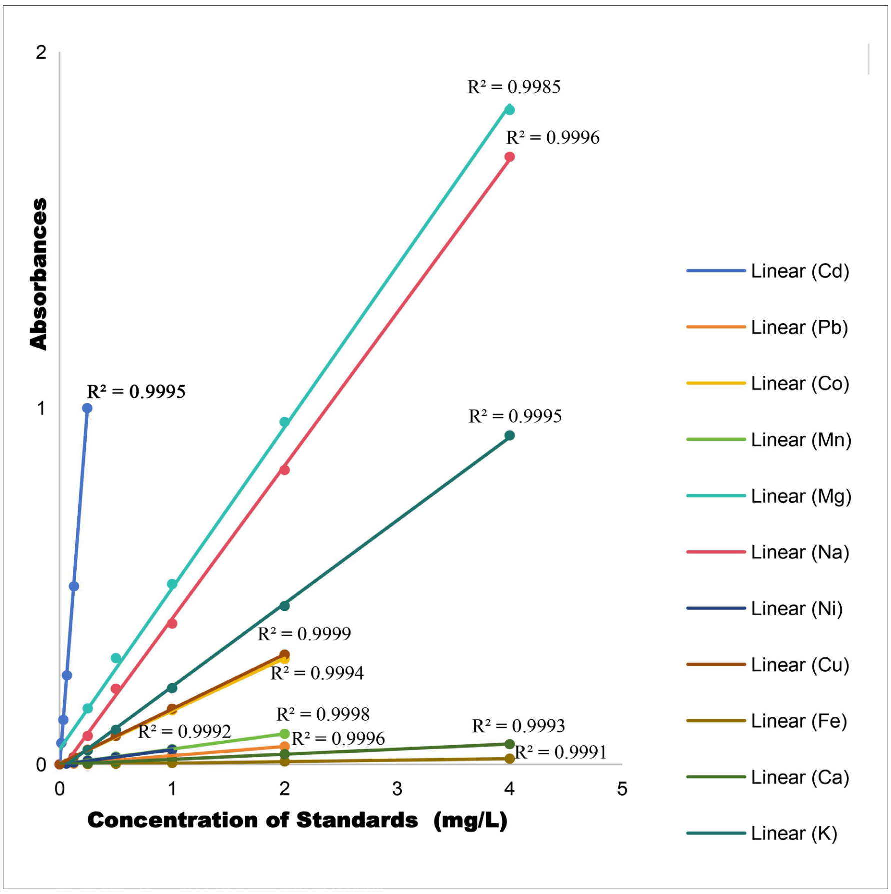

Figure 1. Calibration curve for micro and secondary elements content determination in the samples.

The concentrations of the standard solutions were chosen based on the predictable concentrations of the solutions studied. These must lie within the ranges of the standard solutions. These concentrations were read under the experimental conditions summarized in Table 1.

Table 1. Analytical parameter, technique, specific equipment used and reference standards.

Analytical Parameter	Technique	Specific equipment used	Reference Standards
Calcium	Flame Atomic Absorption Spectrometry	THERMO FISCHER iCE 3000 SERIES AAS	NF EN ISO 7980
Sodium Potassium	Flame Atomic Absorption Spectrometry	THERMO FISCHER iCE 3000 SERIES AAS	NF T 90 - 020
Iron Manganese Magnesium	Flame Atomic Absorption Spectrometry	THERMO FISCHER iCE 3000 SERIES AAS	FD T 90 - 112

Continued

Cadmium	Flame Atomic Absorption Spectrometry	THERMO FISCHER iCE 3000 SERIES AAS	NF EN ISO 5961
Lead Cobalt Copper Nickel	Flame Atomic Absorption Spectrometry	THERMO FISCHER iCE 3000 SERIES AAS	FD T 90 - 112

The spectrometer shows the concentration of the element studied in the samples directly on the computer. These concentrations are expressed in mg/l. However, the actual concentrations (C) of the elements measured in the samples are expressed in mg/kg of dry mass (DM). This concentration was calculated according to the following equation

$$C\left(\text{mg/kg DM}\right)=\frac{\text{Concentration displayed by the spectrometer}\times\text{Filtration volume}}{\text{Mass of sample taken}}. \quad (2)$$

2.5.8. Determination of Calcium Carbonate ($CaCO_3$) by Back Titration

An excess volume of hydrochloric acid was added to the sample to decompose the carbonates. The remaining acid was then analysed by titration of the residual-free solution with a basic solution. The difference represents the acid used to decompose the carbonates. It is worth noting that the acid is neutralized by any base present in the sample, but the result is expressed in $CaCO_3$ equivalent [22].

Expression of the results:

$$C(\text{mg } CaCO_3 \text{ for 0.1 Kg}) = [20 - TN{*}\,V/A]{*}50{*}100/S$$

T: Volume of Sodium hydroxide used as a dose for the sample (ml);
N: Concentration of the Sodium hydroxide;
A: Aliquot (ml);
V: Volume of filtrate (ml);
S: Weight of the sample (g);
1 ml of hydrochloric acid corresponds to 50 mg of calcium carbonate.

3. Results and Discussion

3.1. General Characteristics of Wood Ash

The physico-chemical parameters result of the ash are indicated in **Table 2**. Major and secondary element concentrations are shown in **Figure 2**.

SOPAL eucalyptus wood ash contains an average of 10.21 g of water per kg of dry mass, about 1% of the ash mass. This water corresponds to the humidity probably absorbed in the air by the ash during storage, and to the water added to the ash to extinguish it and reduce its powderiness. The addition of this ash to the soil is likely to boost their water-retention capacity.

The ash has an average pH of 12.97. This very basic pH is explained by the quantity of alkaline elements such as calcium, potassium, sodium and magnesium in the ash (**Figure 2**). This general characteristic of the ash, confirmed by

Table 2. Physico-chemical characteristics.

Parameters	Content
H_2O (g/kg DM)	10.21
pH	12.97
OM (g/kg DM)	42.82
TOC (g/kg DM)	24.84
$CaCO_3$ (g/kg DM)	24.02
EC (mS/Cm)	24.10

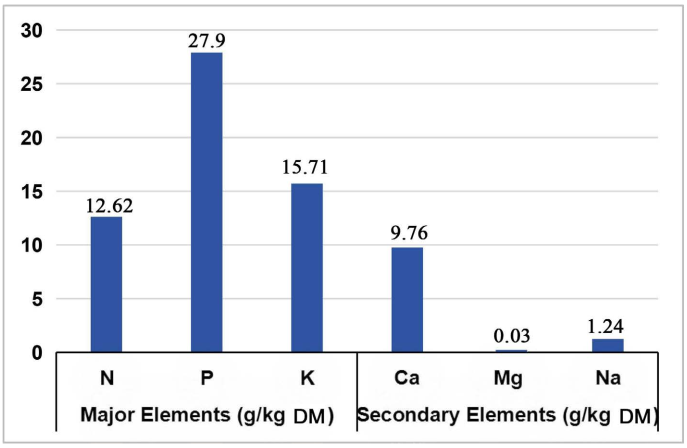

Figure 2. Major and secondary elements content.

Demeyer and al. [8], is a point of great agronomic interest, especially in modifying the pH of acid soils to adapt them to some basophilic crops and to combat heavy metals precipitation in acid soils.

A large proportion of the calcium, about 98%, is in the form of calcium carbonate (Table 2), while the remaining is in the much more soluble form of calcium hydroxide (Figure 2), since in the presence of moisture the quicklime (CaO) in the ash is transformed into hydroxide; the same applies to potassium [9]. Since calcium carbonate (calcium lime), accounts for the highest proportion of the calcium compounds in the ash, it would enable the latter to balance slowly the pH of agricultural soils without excess, due to its content of carbonate ions (CO_3^{2-}) rather than hydroxide ions. Moreover, since calcium carbonate is not easily soluble in water and only reacts in an acidic medium ($CaCO_3 + 2H^+ \rightarrow Ca^{2+} + CO_2 + H_2O$), its effect on the soil would be much long-lasting, enabling farmers to considerably reduce their spreading frequency and save on ash.

The electrical conductivity (EC) of the ash (24.10 mS/Cm) is mainly due to its alkalinity, *i.e.* the presence of soluble elements rich in alkaline ions (Ca^{2+}, K^+, Mg^{2+}, Na^+) directly available to plants and soil.

A large proportion of the organic matter (OM) and organic carbon (OC) in wood burns during combustion. Thus, SOPAL ash contains about 4% of OM and of 2% OC. These quantities of organic matter and organic carbon are about three times greater than those of Enerbois ashes [12] and, if spread over the long term, can contribute significantly in enriching the soil and crops with organic matter. The presence of organic matter and organic carbon in ash is due partly to the charcoal debris present in the ash and partly to the presence of micro-organisms as a result of humidity.

3.2. Content of Major and Secondary Nutrients

The contents of major (N, P, K) and secondary (Ca, Mg, Na) nutrients in the ash are compiled then compared on with some results from the literature as indicated in **Figure 3**.

SOPAL wood ash contains more calcium and potassium than other alkaline elements such as magnesium and sodium (**Figure 2**). Comparatively most of the chemical elements it contains and with data from the literature, the SOPAL wood ash we recovered is very rich in major elements such as nitrogen (about 1.3% of dry mass) and phosphorus (about 2.8% of dry mass, in the form of P_2O_5). Potassium (about 1.6% of dry mass). However, it has a high concentration compared to other chemical elements in the ash, and a low concentration compared to data from the literature (**Figure 3**). All these differences in the NPK content of the SOPAL ash compared with data from literature are probably linked either to the nature of the wood, its origin or both factors combined.

3.3. Microelements Content

The microelements (Fe, Mn, Co, Cd, Pb, Cu, Ni) in the ash and in some samples of eucalyptus wood from the refinery were analysed using an atomic absorption spectrophotometer, and the results are shown in **Table 3**.

Analyses carried out on the metallic trace elements in our ash and data from the literature (**Table 3**) showed that iron and manganese are the most abundant elements. Iron and manganese contents are respectively 23 and 49 times higher in the ash than in the wood (**Table 3**). The greater iron content in the ash can be explained probably by the wear and tear of the furnace parts (**Image 7** and **Image 8**), and by the concentration effect of microelements in the ash during wood incineration [9] [10]. The same concentration effects probably explain the difference in metal content in the ash and the wood. According to studies carried out by Hébert and Breton [9], aluminium, iron and manganese are the metals most frequently found in ash. Metals such as zinc, lead, mercury and cadmium volatilize and concentrate more in fly-ash, unlike copper, arsenic and chromium [11]. These results demonstrate the low Zn, Pb, Hg and Cd content of SOPAL ash, and at the same time prove that the copper content is almost entirely derived from the wood.

Metallic Trace Elements (MTE) are less soluble in a basic medium [9]. This hypothesis was proved by Agwaramgbo and al. [23] and also by Sinaj and Maltas [24] for Enerbois ashes by the solubility analysis of some MTE (Co, Mn, Cd, Cu, Pb, Zn and Fe). Thus, according to Sinaj and Maltas, no phytotoxicity due to MTE is expected in the short term after the application of the ashes since they are normally found in forms much less absorbable by plants. However, in the

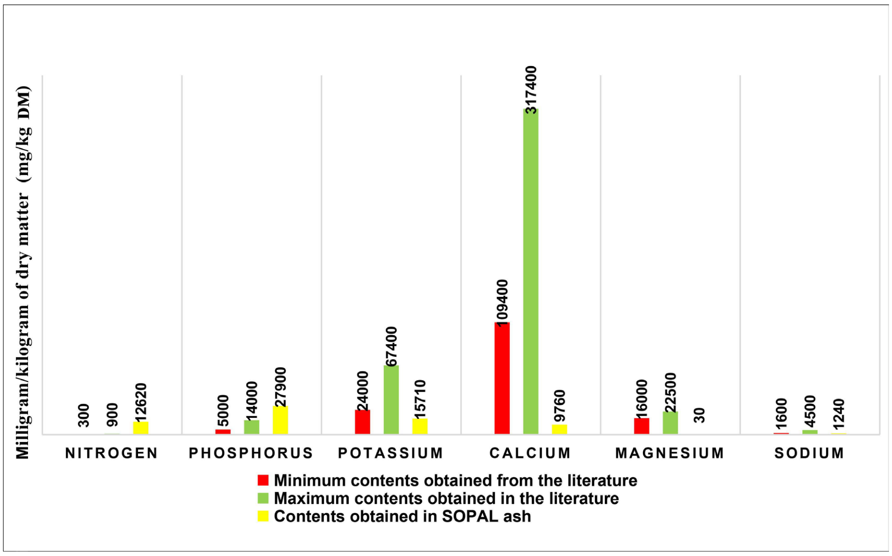

Figure 3. Major and Secondary Content of SOPAL Ash and Literature (Demeyer and al. 2001, Hebert and Breton 2008, Maltas and Sinaj 2014).

Table 3. Total microelement content of the ash and wood.

Microelements	Total Content (mg/kg DM)		
	The SOPAL Wood Ash	SOPAL Wood Sawdust	Ash from the littérature [8] [9] [12]
Fe	1491.31	65.35	3300 - 19,500
Mn	7819.22	160.12	3470 - 8160
Co	0.19	0.03	4 - 10
Cd	0.10	0.08	<0.58 - 21
Pb	1.14	0.55	<22 - 130
Cu	19.77	0.69	74 - 145
Ni	12.82	0.55	12 - 56

long term, the question of phytotoxicity of ashes remains due to the possible reacidification of soils [8]. Such a risk is unlikely on agricultural soil, because to avoid any drop in the yield, the farmer generally intervenes before the soil becomes too acidic [24]. Thus, according to Hébert and Breton [9], the risks related to MTE in ashes are considered negligible in the short and medium terms and improbable in the long run when liming is practiced at agronomic doses. SOPAL ash having the lowest MTE content compared to data from the literature (Table 3), should not pose any phytotoxic problem.

3.4. Metallic Trace Elements Contents

The spreading of the ash has to be regulated. The regulations mostly set the dose to be spread based on the heavy metal contents limit. The contents may vary from country to country. By comparing the heavy metal contents of the SOPAL ash to the thresholds set by Switzerland, France, Germany, Austria, Finland and

Canada, we see that the contents of trace metal elements (Cd, Pb, Cu, Ni) of the ash are considerably low (**Table 4**).

Table 4. Metallic trace element contents (mg/kg DM) of the SOPAL Ash.

	Data from the literature [10] [11]						SOPAL
ETM	Finland	Austria	Switzerland	Canada	Germany	France	
Cr		250		120	2	150	
Ni	100	100	30	32	80	50	12.82
Zn	1500	1500	400	220	1000	300	
Cu	600	250	100	100	70	100	19.77
Cd	3	8	1	1.6	1.5	2	0.1
Pb	150	100	120	60	150	100	1.14
Hg	2		1	0.5	1	1	
As	50	20		14	40		

Hence, based on the thresholds set by these six countries, we can say that SOPAL's wood ash can be spread not only on agricultural land, but also on forests land since the thresholds set for spreading on agricultural land are far below the thresholds set for spreading on forest land [10].

Wood ash has effect on both the soil and plants. Thus, according to Hanssen and al [25], a mixture of wood ash and nitrogen has a more significant effect in the short-term and long-term growth of forest trees than fertilization with wood ash alone. The use of wood ash can contribute to improving the structural characteristics of plants [26] and the reduction of N_2O (greenhouse gas) emissions from soils during nitrification or denitrification processes in the soils [27] [28]. SOPAL wood ash is very alkaline, and has a significant content of potassium, calcium and especially phosphorus and nitrogen. This ash has alkaline-rich properties, therefore can be used to fertilise alkalinophilic crops such as barley, beets, beans and alfalfa [24]. The contents of N (1.3%), P (2.8%), K (1.6%) and Ca (0.9%) in the ash give it a high fertilizing property. The potentials of this ash can be boosted by combining it with other materials such as fertilizer [28], sanitized human excrement [29], which will reduce the use of chemical fertilizer. The ash can also be mixed with mature compost to boost the performance of the compost [30] [31]. SOPAL wood ash has a high manganese content (**Table 3**) therefore suitable for flower gardening [32].

According to Sinaj and Maltas [24], wood ash can be used as a compost activator, because the addition of alkaline elements stimulates humification by increasing the microbial population and their activity. According to the latter, this property of ash becomes particularly interesting when the compost is made of plant residues rich in lignin and slow to decay. Thus, wood ash acts as a catalyst in the composting process by accelerating it and facilitating the digestion of woody materials. Wood ash not only enriches the compost with minerals, but also increases the alkalinity of the compost, thus reducing the quantity of compost to be spread for liming acidic soils. Amendments resulting from co-composting with ash can help to remove heavy metals from the soil, because these amendments in another way, often have high pH values, which influence the solubility of these heavy metals (Co, Mn, Cd, Cu, Pb, Zn and Fe) thus making them unavailable for plants [10]. In another way, the organic materials in the compost can form organometallic complexes with the heavy metals in the ash, thus increasing their unavailability for plants.

4. Conclusions

Hence, we noted after analysis, that SOPAL wood ash presents obvious potentials in favor of farming because, on the one hand, its TME contents are well below the thresholds set by six countries (France, Austria, Germany, Finland, Canada, Switzerland) and present very low values compared to the data recorded in the literature.

On the other hand, it presents interesting agronomic characteristics such as a high pH (12.97) which gives it, not only a liming character but also a lasting effect when put to the soil, because of its high calcium carbonate content compared to quicklime (98%). For its high alkaline (pH) quality, SOPAL ash can be used as a powerful activator in co-composting and thereby accelerate the process, especially when the compost is made up of woody materials. It has a high manganese content which is suitable for flower gardening. The non-negligible contents of potassium (15.71 g/kg DM), calcium (9.76 g/kg DM) and especially phosphorus (27 g/kg DM) and nitrogen (12.62 g/kg DM) add more agronomic values to the SOPAL wood ash.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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