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Design and Application of an Innovative Composting Unit for the Effective Treatment of Sludge and other Biodegradable Organic Waste in Morocco

MOROCOMP (*LIFE TCY05/MA000141*)



Task 3: Development of sludge aerobic composting – Optimization of the operation of the pilot composting system

Deliverable 13:

**Report on the physicochemical analysis of sludge and other biodegradable waste and
additives**

Report on the optimum operating conditions for the compost unit

Report concerning the evaluation of compost products (level A)



composting
MOROCOMP

April 2008

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Summary

Within the framework of MOROCOMP project, an innovative composting system for the effective treatment of sludge and other biodegradable organic waste was designed, constructed, set up and operated at the experimental station SEMVA (Station Expérimentale de Mise en Valeur Agricole) in Zemamra in the greater area of Doukkala region in Morocco. This report aims to develop sludge composting and co-composting processes using various waste streams and to optimise the operation of the system.

To determine the optimal course of composting four different composting trials have been performed using a variety of waste such as primary and secondary sludge from the WWTP of El Jadida city in Morocco, sugar beet leaves, straw residues, sheep and cow manures in various ratios and under different operational conditions. Composting trials have also been performed using effective additives such as zeolite and perlite for the treatment of the aforementioned waste in order is to assist composting process without participating in the biodegradation process. The selection of the experimental set-up was determined according to the feedstock material and its characteristics while at the end of each trial valuable feedback was obtained for the optimization of the next composting trials. The temperature, moisture and oxygen content of the substrate was closely monitored on a daily bases during composting while for the evaluation of the processes complete physicochemical analyses have been performed (i) to the raw material that was used as feedstock to the in-vessel composting bioreactor (ii) to the leachates generated during composting (iii) to the substrate throughout the duration of the composting processes and finally (iv) to the derived product resulting from each composting trial.

With respect to the characteristics of the raw material used, sludges acquire relatively low solid content ranging from 21.45% to 32.16% on dry basis due to the absence of dewatering installations near the area where sludges were produced. This problem was overcome with the addition of dried Biodegradable Organic Waste (BOW). In addition, the feedstock material is rich in nutrients content and acquires low heavy metals concentration conditions which favor the production of a good quality compost at the end of each trial.

The temperature profiles of the composting processes, are typical of a well operated composting process since substrates pass from an initial mesophilic stage to a thermophilic one and then to a second mesophilic phase indicating that the processes is being well developed. In regard to the moisture content the substrates is maintained within optimal levels (40% to 60%) for the majority of compost duration mainly through the hydration system. The oxygen level is successfully controlled and maintained at high levels (>9%) in all four trials ensuring that adequate oxygen is being diffused within the mass of the substrate. The successful control of aeration, hydration and temperature is performed by the control system that constitutes an essential component of the bioreactor.

Leachate is produced from moisture extraction of the substrate mainly due to its hydration and partly by the molecular water resulting from the biooxidation of the organic matter. In all trials the leachates produced contain high quantities of biodegradable organic matter expressed as BOD₅. The BOD₅ concentration varies from 72,705mg/L to 50,984mg/L at the initial stage of composting and then gradually decreases leading to BOD₅ values at the end of the process that range from 8,958mg/L to 16,757mg/L. Also ammonium and nitrates concentrations are very high much higher than the limits set for wastewater disposal. Heavy metals concentration are also present in leachates but much higher quantities are associated with the substrate.

With respect to the substrate chemical characteristics the pH values present an initial increase from near neutral to alkaline values while at the end of the process pH acquires again near neutral values indicating that the composting process is towards the end. The initial C:N ratios are lower than the suggested optimal (20 to 40) due to the origin of the raw material ranging from 8.07 to 19.61 and as composting is kept developing the ratio significantly decreases. The evolution of ammonium and nitrates concentration confirms the stimulation effect of nitrification and that oxidizing factors persist during the processes.

The C:N ratios of the produced composts reflect to a good quality compost since they acquire ratios lower than 12 which is considered as a very good value for finished composts. The nutrients content for all trials indicates that the macroelements quantity

of composts is sufficient for agricultural applications while the ammonium to nitrates ratios ranging from 0.09 to 0.24 are within the suggested limits of fully mature composts. The analysis on heavy metals shows that their concentration is quite low within the suggested EU limits and that only a low percentage ranging from 1.4% to 6.2% of the various metals is available in dissolved forms. Finally the elevated temperatures that occur in all four trials lead to the elimination of the pathogenic microorganisms that are present in the raw material prior to the initiation of composting processes.

1. Introduction to composting process

Composting is the aerobic, or oxygen requiring, decomposition of organic materials by micro-organisms under *controlled* conditions. During composting the microorganisms consume oxygen while feeding on organic matter. This generates heat, carbon dioxide and water vapour, which are released into the atmosphere. Composting reduces both the volume and mass of the raw materials while transforming them into a stable organic fertiliser or soil conditioner. Composting can occur at a rapid rate when optimum conditions that encourage the growth of micro-organisms are established and maintained. As mentioned composting is a *controlled* aerobic (oxygen-using) biological decomposition of most organic (biologically derived carbon-containing) solid matter and that differentiates the process from the natural occurring decomposition. Nevertheless, the biochemical process in composting and in the natural decomposition of the organic matter is the same. The composting process includes two major phases. The first one, called the “active phase”, mainly develops degrading reactions. Dissolved organic matter is used as carbon and energy source by microorganisms for their metabolism. During the second phase of the composting process, called the “curing phase”, organic macromolecules such as humic substances are synthesised. All reactions are based on numerous biological, thermal and physicochemical phenomena and involve oxygen consumption, as well as heat, water and carbon dioxide production. Figure 1 shows a schematic diagram for composting process.

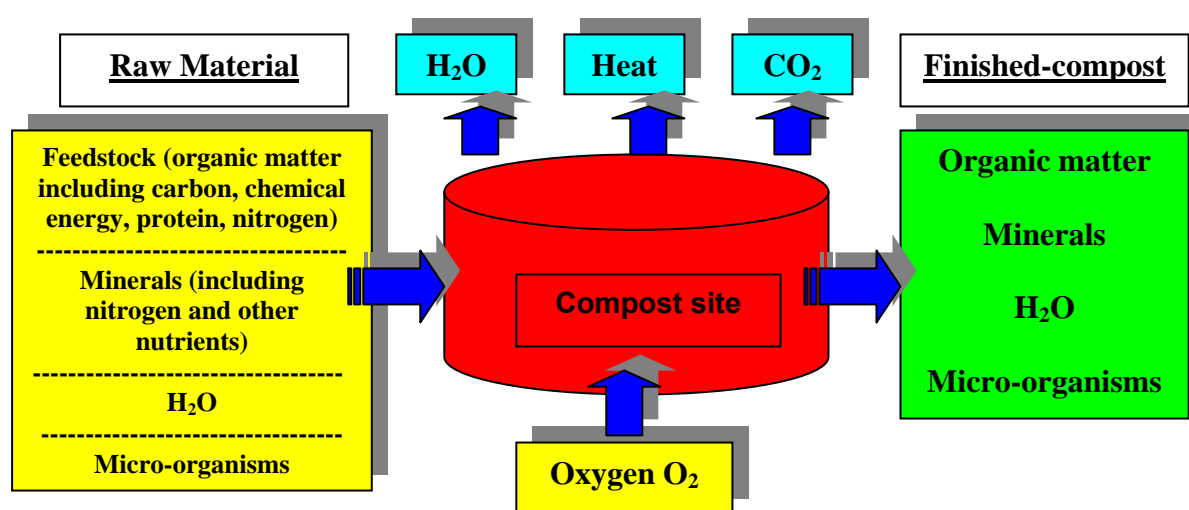


Figure 1: A schematic diagram of composting process [1]

2. Factors affecting the composting process

The factors affecting the composting process include: the physical and chemical properties of the raw material, the level of oxygen, the moisture content, the temperature and the time over which the composting process takes place. These factors are outlined in the following sections.

2.1. Oxygen and aeration

The readily degradable components of the raw materials are metabolised during the initial period of composting. Therefore the production of heat and the need for oxygen are greatest at the early stages and then they steadily decrease. Without a constant supply of oxygen, the process will slow down. Approximately a 5% minimum concentration of oxygen is required within the pore spaces (given air contains about 21% oxygen). If there is insufficient oxygen, the process can become anaerobic. Anaerobic decomposition involves a different set of micro-organisms and different biochemical reactions. Less heat is generated and the anaerobic processes are potentially less efficient than the aerobic ones. They can generate various substances and intermediate compounds such as methane, organic acids and hydrogen sulphide. Many of these compounds have strong odours and safety concerns. Although intermediate compounds, such as organic acids, are formed during aerobic decomposition, they continue to decompose when oxygen is available. Under anaerobic conditions, the intermediate compounds can accumulate. A constant supply of oxygen will give the aerobic microorganisms an advantage over the anaerobic micro-organisms. The existence of aerobic conditions is very important in avoiding the production of offensive odours.

Aeration is the process of providing oxygen into the composting material, as well as the mean to remove water vapour, gases and heat trapped within the materials. Temperature often is an indicator that determines how much and how often aeration is required, given that the required rate of aeration for heat removal can be much greater than that for supplying oxygen. Similarly the aeration rate required to reduce the moisture content is normally greater than that required for supplying oxygen, but less than the heat removal rate.

2.2. Particle size, porosity, structure and texture

The physical properties of the raw materials affect the composting process by their influence on aeration. They can be adjusted by the selection and mixing of raw materials. Any materials added to adjust these properties are known as bulking agents. Texture is the characteristic that describes the available surface area for aerobic micro-organism activity. The majority of aerobic decomposition occurs on the surface of particles because oxygen moves readily as a gas through pore spaces, but much slower through liquid and solid portions of the particles. For most raw materials and composting applications, an acceptable porosity and structure can be achieved if the moisture content is less than 65%. Since the amount of surface area increases with smaller particle size, the rate of aerobic decomposition also increases with smaller particle size to a certain extent. Smaller particles reduce the effective porosity and therefore a compromise is needed. An average particle size of 10mm to 50mm generally produces the best results. However certain composting methods that do not include a turning process require a more robust physical structure to resist settling, so larger particles are necessary (greater than 50mm).

2.3. Nutrients and the Carbon to Nitrogen ratio

Microorganisms involved in composting require the following main nutrients: potassium (K), nitrogen (N), phosphorous (P) and carbon (C). Nitrogen, potassium and phosphorous are also the primary nutrients for plants; so their concentrations also influence the potential value of the composted organic material. The majority of organic materials contain ample quantities of nutrients. The amounts of carbon or nitrogen are therefore the substances most likely to affect the composting process by their presence in insufficient or excessive quantities. Nitrogen is used by micro-organisms for protein production and carbon is used for energy and growth. Generally speaking, biological organisms need about 25 times more carbon than nitrogen. The ratio of carbon to nitrogen is referred to as the C:N ratio. Raw materials mixed to provide a C:N ratio of 25:1 to 30:1 are generally accepted as ideal for active composting, although ratios from 20:1 up to 40:1 can give good composting results. Low C:N ratios of below 20:1 allow the carbon to be fully utilised without stabilising the nitrogen, which may be lost as ammonia or nitrous oxide causing potential odour problems. Ratios of composting materials having carbon content higher than 40:1

require longer composting periods while the excess carbon is used by the micro-organisms.

The rate at which carbon compounds decompose must also be considered. If the carbon is in a form that is difficult to decompose, the composting rate may be slower (e.g. wood waste). Since decomposition occurs on particle surfaces, degradability can be improved by reducing the particle size as long as porosity is not a problem (see above). If required, the C:N ratio can be adjusted to increase the proportion of carbon to compensate for the presence of poorly degradable sources of carbon. This may, however, results in a longer composting time period.

2.4. pH

The composting process is relatively insensitive to pH within the range commonly found in mixtures of organic materials. The composting process can work over a wide spectrum of pH values, but a range of between 6.0 and 8.5 is preferred [2, 3, 4, 5, 6]. Although the composting process will be less effective at the extreme ranges of 5.5 and 9.0 it will still work. A pH above 8.5 encourages the conversion of nitrogen compounds to ammonia, which further adds to the alkalinity. Adjustment of the pH downward below 8.0 to reduce the ammonia loss is possible by using additives. As decomposition occurs the composition of the raw materials and the associated pH change. However, irrespective of the pH of the starting raw materials, the composting process yields a final composted organic material with a stable pH that is close to neutral (7.0).

2.5. Moisture

Moisture supports the metabolic processes of the micro-organisms. Water is the medium for chemical reactions, transportation of nutrients and allows the microorganisms to move about. Biological activity ceases below 15% moisture content and in theory activity is optimal when materials are saturated. Generally moisture content of between 40% and 65% should be maintained. At moisture content of below 40%, micro-organism activity will continue but at a slower rate and above 65% water will displace much of the air in the pore spaces of the composting material.

This will limit the movement of air and lead to anaerobic conditions. Moisture content should be above 40% at the starting point, as it will generally decrease as composting proceeds. Therefore if the moisture content falls below 40%, water should be added to maintain optimum conditions.

During composting the moisture levels change as water evaporates and usually there is a need for water addition. In many composting cases more water evaporates than is added so the moisture tends to decrease as the composting proceeds. Moisture levels should be maintained so that materials are thoroughly wetted but without being waterlogged.

The moisture content range of 40-65% works well for most materials. The upper limit for moisture content is dependent on the absorbency and porosity of the raw materials. A mixture with highly absorbent materials (e.g. dry grass) may need to be maintained well above 40% moisture in order to support rapid decomposition. Materials should not be allowed to dry out below 40% moisture content, as this will increase the chance of damaging high temperatures or even spontaneous combustion.

2.6. Temperature

Composting takes place within two temperature ranges known as mesophilic ($\approx 35^{\circ}\text{C}$ - 40°C) and thermophilic ($>40^{\circ}\text{C}$). It is generally accepted that maintaining temperatures between 43°C and 65°C allows for effective composting. The thermophilic temperatures are favoured in the composting materials, because they destroy more pathogens, weed seeds and fly larvae. A temperature of 55°C should kill human pathogens and most plant pathogens as well. Micro-organism activity during composting releases large amounts of energy in the form of heat. This heat accumulates due to the self-insulating qualities of the compost material and causes the temperature to rise. At the same time water evaporates and water vapour and warm gases are vented. Turning and aeration accelerate the heat loss and is used to maintain temperatures within the desired range.

When temperatures rise above 60°C the micro-organisms suffer the effects of high temperatures and the process slows down. Temperatures can continue to rise up to

above 70°C due to insulation effects and on-going microbial activity. At these temperatures many micro-organisms die or become dormant and the process effectively stops until the micro-organisms can recover. Turning or aerating the substrate when it approaches 60°C can prevent this. If thermal kill does occur, the substrate should be re-mixed using material from biologically active batches.

2.7. Retention time

The time period required to produce a composted organic material depends on many factors, including user needs, moisture, temperature and the used raw materials. Factors, which may slow the process down, include a high C:N ratio, low temperature, insufficient aeration, lack of moisture, large particles and high percentages of resistant materials such as wood. If the composted organic material does not need to be completely stable for specific agricultural or biological applications, it can be matured and finished where it is applied to land. The composting period is often extended for composted organic products, which need to be stable or particularly dry.

3. The need for sludge and BOW composting

The main objective of MOROCOMP project is to develop and establish an innovative composting system for the treatment and reuse of sludge and other biodegradable organic waste (BOW). The environmentally sound and commercially feasible management of sewage sludge is a major issue that all European countries confront. Sewage sludge arises from the processes of wastewater treatment and represents one of the top priority waste streams. The most important reason that sludge generation, disposal and recycling has been of particular concern lies in the fact that pollutants contained in sewage sludge may impose potential risks on human health and on the environment when applied to agricultural land without controlling its qualitative and quantitative characteristics.

The selected substrate that was used as feedstock material into the bioreactor comprised of primary sludge, green waste and manure of different origin (sheep, cow). Sewage sludge was selected to be the primary raw composting material since

sludge treatment is an immense problem that Moroccan authorities have to cope with. More specifically, the annual volume of urban wastewater has increased from 48 million m³ in 1960 to 500 million m³ in 1999. It is estimated that this amount will reach 900 million m³ in 2020. This significant increase is due to population growth, which is estimated at 5% per year. In Morocco, untreated wastewater is discharged in the water recipients. Approximately 60% is discharged in the sea which poses a real health hazard as has been shown by recent studies [7] while the rest is either discharged in the surface waters or reused for the irrigation 7000ha of land [8]. It should also be mentioned that EU has initiated the horizon 2020 programme for the de-pollution of the Mediterranean Sea by 2020 [9]. This means that effluents should not be discharged to sea recipients unless they are treated. So Morocco will have to cope with the continuous increase of sludge in a sustainable manner.

Composting process is one of the best waste management methods as far as sustainability issues are concerned, to stabilise organic waste including sewage sludge. Sewage sludge provides labile organic matter in sufficient quantities to stimulate soil microorganisms. This kind of organic residue improves the soil's physical characteristics; increasing soil sponginess, water holding capacity and the percentage of stable aggregates. They also improve the nutritional quality of soil and most importantly increase the content of the labile carbon fractions, which acts as a catalyst for the microorganisms, thus improving the soil's potential fertility and stimulating the biogeochemical cycles of the most important elements. Therefore the use of sludge as a composting material can be of significant value if the end product is of high or even moderate quality.

Biodegradable Organic Waste or Biodegradable Waste is termed as “... *any waste that can undergo anaerobic or aerobic decomposition, such as food waste, garden waste, paper and paperboard*” [10]. BOW can be biologically decomposed into a stabilized product and among many waste it includes agricultural waste, green waste, animal manure etc. Therefore composting contributes in diverting organic wastes from landfill sites, conserving landfill space, reducing the production of leachate and methane gas while at the same time it enables recovering and recycling nutrient content of BOW. It is worth mentioning that EU directive 1999/31/EEC¹ sets the limits for the landfilling of organic waste in order to assist the minimization of green

¹ EU Directive 1999/31/EEC on the landfill of waste

house gas emissions. In addition Morocco's economy is heavily dependent on agricultural and animal husbandry practices therefore the increased amounts of green waste and animal manure that is produced, raise a point of concern with respect to the management and treatment of these wastes.

The experimental composting trials developed during the 3rd Task of MOROCOMP project aim to present the compost quality characteristics and the effects of the unit's operational conditions on the efficiency of the process. Therefore for each of the composting trials that have been developed a series of steps have been performed.

4. Methodology and experimental procedures

The development of aerobic composting processes using sludge, other BOW (green waste and manure) and effective additives (zeolite, perlite) were studied in this programme. To justify the successful implementation of the development of effective composting and co-composting processes and optimisation of the system various experiments were carried out. These experiments were focused on the compost quality characterization and on the effects of the operational conditions on the efficiency of the process, as well as on the end product quality.

The achieved quality of the end product was the criterion for evaluating the composting process. For this purpose complete physicochemical analysis of the end product resulted from extended series of experiments that took place. The parameters that needed to be measured were the total carbon, total nitrogen, total and available phosphorous, total water content, pH, total and available potassium, sodium, magnesium, manganese and calcium, heavy metals content and speciation, biological and microbiological activity analyses.

Several composting trials have been performed throughout the duration of the 3rd Task of the MOROCOMP project using the composting system that has been designed and constructed in Task 2. Initially the raw materials that had been used for composting were selected and analysed. These materials included primary sewage sludge from the city of El Jadida, sugar beet leaves, straw residues, sheep and cow manures in various ratios.

Due to the unavailability of the UWWTP in El Jadida at the time of the execution of the project to provide us with secondary sludge, no composting trials have been performed using secondary sludge from an UWWTP as raw material for composting. Although there were other treatment plants operating outside the city of El Jadida that could provide us with sufficient amount of secondary sludge, the long distance as well as the high transport cost were prohibitive to apply this material into the bioreactor from those plants. Instead of secondary sludge from the UWWTP in El Jadida it was considered appropriate to perform a composting trial, the 4th composting trial, using secondary sludge from a food industry.

Although in the initial proposal it was stated that the composting system would also treat sludge generated from Potable Water Refineries (PWR) in Morocco, it was not considered appropriate to use that type of sludge into the composting system since the high content of inert material, mainly sand and minerals, was qualitatively inappropriate for the composting process. Substantial amount of sand and minerals existed in the primary sludge.

Furthermore, due to the high moisture content of primary sludge it was considered appropriate not to use primary sludge individually but to mix it with green waste which operate as excellent amendments and bulking agents. The high moisture content of sludge arises from the fact that there were not dewatering installations in the region where primary sludge was produced. In addition, rarely will an organic material have all the characteristics needed for efficient composting, so other materials² must be blended to achieve the desired characteristics. Green waste can adjust the mixture's moisture content the carbon to nitrogen ratio and the texture of the substrate while it provides pore spaces for air movement. Therefore green waste makes a good quality structuring agent to be mixed with sewage sludge for composting.

Green waste comprised of sugar beet leaves and straw provided by Moroccan farmers. Since sugar beet and wheat are the primary cultivations that are being practiced in

² amendments or bulking agents

Doukkala region³, sugar beet leaves and straw residues were considered appropriate to be used as an input composting material due to their high availability and negligible cost. Furthermore manure of different origin was also used as raw material for compost. Sheep and cow manures were the types of manure used in the composting trials due to their high availability, low heavy metal content and the absence of other hazardous substances. Furthermore manures high nitrogen and phosphorous content constitutes a basic nutrient component for plant growth.

4.1. Methods for sampling and analysis of the raw material

The characterisation of the raw material that was used as a feedstock to the in-vessel composting bioreactor is essential for the evaluation of the aerobic composting processes and thus to the evaluation of the end products. The feedstock determines a priori the physical chemical and biological conditions involved in composting processes. The rate at which materials compost and the qualities of the final composted organic material are largely dependent on the initial selection and mixing of raw materials.

For each examined raw material, six representative samples were being collected. The final sample was formed after stirring and mixing the six same weighted samples together to form a homogeneous material. From this homogeneous material six separate samples were selected from which a series of parameters were evaluated. The average value of each parameter was calculated from the average value of the separate samples. The examined parameters of the raw material are listed in Table 1. Table 1 also presents the standard methods followed for testing and evaluating compost and composting feedstock material. These standard methods have been employed in order to analyse and evaluate the initial material that was selected to be composted as well as the end product that resulted after the composting process has been completed.

³ greater area of El Jadida

Table 1: Parameters evaluated the standard methods used and their references

Test Parameter	Method	Reference
% Solids, % Moisture	Method 2540B (<i>Dried at 105 C</i>)	Standard Methods for the Examination of Water and Wastewater. 1992. 18th Edition, American Public Health Association, 1015 Fifteenth Street, NW, Washington , DC 20005.
pH	TMECC 4.11-A (<i>1:5 w:w slurry</i>)	Test Methods for the Examination of Composting and Compost, USDA and U.S. Composting Council. 2002.
Total Carbon	TMECC 4.02-D (<i>Combustion</i>)	Test Methods for the Examination of Composting and Compost, USDA and U.S. Composting Council. 2002.
Total Nitrogen	TMECC 4.02-D (<i>Combustion</i>)	Test Methods for the Examination of Composting and Compost, USDA and U.S. Composting Council. 2002.
Ammonium-N	TMECC 4.02-C (<i>1:5 w:w slurry</i>)	Test Methods for the Examination of Composting and Compost, USDA and U.S. Composting Council. 2002.
Nitrate-N	TMECC 4.02-D (<i>1:5 w:w slurry</i>)	Test Methods for the Examination of Composting and Compost, USDA and U.S. Composting Council. 2002.
Phosphorus	TMECC 4.03-A	Test Methods for the Examination of Composting and Compost, USDA and U.S. Composting Council. 2002.
Potassium	TMECC 4.04-A	Test Methods for the Examination of Composting and Compost, USDA and U.S. Composting Council. 2002.
Calcium	TMECC 4.05-Ca	Test Methods for the Examination of Composting and Compost, USDA and U.S. Composting Council. 2002.
Manganese	TMECC 4.05-Mn	Test Methods for the Examination of Composting and Compost, USDA and U.S. Composting Council. 2002.

Heavy Metals		
Cadmium	TMECC 4.06-Cd	Test Methods for the Examination of Composting and Compost, USDA and U.S. Composting Council. 2002.
Chromium	TMECC 4.06-Cr	Test Methods for the Examination of Composting and Compost, USDA and U.S. Composting Council. 2002.
Copper	TMECC 4.05-Cu	Test Methods for the Examination of Composting and Compost, USDA and U.S. Composting Council. 2002.
Nickel	TMECC 4.06-Ni	Test Methods for the Examination of Composting and Compost, USDA and U.S. Composting Council. 2002.
Lead	TMECC 4.06-Pb	Test Methods for the Examination of Composting and Compost, USDA and U.S. Composting Council. 2002.
Zinc	TMECC 4.06-Zn	Test Methods for the Examination of Composting and Compost, USDA and U.S. Composting Council. 2002.
Heavy Metals Speciation	40CFR 261.24	US EPA Extraction Procedure Toxicity Test Federal Register
Pathogens Testing		
Total Coliforms	TMECC 07.01-A	Test Methods for the Examination of Composting and Compost, USDA and U.S. Composting Council. 2002.
Faecal Coliforms	TMECC 07.01-B	Test Methods for the Examination of Composting and Compost, USDA and U.S. Composting Council. 2002.
Helminths	TMECC 07.04	Test Methods for the Examination of Composting and Compost, USDA and U.S. Composting Council. 2002.

4.2. Composting mixtures

The chemical composition of the substrate as well as the particle size, porosity, structure and texture of the raw composting material significantly affect composting processes. The easily hydrolyzed chemical compounds accelerate composting while the surface to volume ratio of particles influences significantly the composting conditions.

The primary sludge from El Jadida city, that was used as a primary raw composting material, did not undergo dewatering and the addition of green waste, acquiring low water content, was considered appropriate in order to balance the level of the substrate's water level. Feedstock with different moisture holding capacities can be blended to achieve ideal moisture content. Another important reason for mixing green waste with sludge is that green waste constitutes a good bulking material which modifies the physical properties of the substrate by lowering the moisture content, providing structural support, increasing porosity, and favoring aeration. In addition, green waste acquire notable amount of nutrient compounds thus providing essential minerals, mainly nitrogen and phosphorous, to the substrate.

Manure of different origin (sheep and cow) was also used in the composting trials since the high availability and organic content, the low heavy metal content and the absence of other hazardous substances make manure excellent composting material. Furthermore manure acquires high nitrogen and phosphorous content which are basic nutrient components for plant growth.

Additives such as zeolite and perlite were obtained from Moroccan local market. Zeolite was used as additive in order to examine primarily the heavy metals removal from the substrate and secondly its capability to adsorb the volatilized ammonia during composting. The importance of the later lies in the fact that nitrogen losses, which occur mainly during the initial stages of composting, result from ammonia volatilization. High losses of ammonia nitrogen represent a waste of a valuable resource and reduce the agronomic value of the end-product. The importance of perlite use as an additive lies in the fact that it modifies the physical properties of the substrate (e.g. structural support, porosity, aeration) to promote composting by

increasing the void volume of the substrate without involving into the biochemical process of composting due to its inert properties.

4.2.1. Pretreatment and preparation of the composting raw material

Green waste was being shredded using a shredding device in order to minimize the size of waste and to increase the porosity of the substrate and to ease the agitation of the composting raw material. All the materials used as feedstock to supply the bioreactor were weighted, according to the composting recipe of each composting trial, and packed in order to be ready to be fed into the bioreactor.

4.2.2. Feeding the in-vessel bioreactor

The feeding of the bioreactor was performed via the conveyor belt on which the feedstock was loaded manually using shovels.

4.3. Composting process control

Composting process control involves all the actions that had been planned and carried out with respect to the operational conditions of the in-vessel bioreactor.

4.3.1. Programming the plc of the in-vessel bioreactor

The operation of the agitation, hydration and aeration systems is being performed by the PLC (Programming Logic Controller) automatic control system. These operations can also be performed manually. The specifications of each operation are given below:

Programming the frequency and duration of the substrate's agitation

The electric monitor can operate X times per day for a duration of Y minutes per hour. The maximum set values are $X_{\max} = 12$ times/day and $Y_{\max} = 15$ minutes while the minimum ones are $X_{\min} = 0$ times/day and $Y_{\min} = 0$ minutes.

Programming the frequency and duration of the substrate's aeration

The ventilator system can operate X times per day for a duration of Y minutes per hour. The maximum set values are $X_{\max} = 24$ times/day and $Y_{\max} = 10$ minutes while the minimum ones are $X_{\min} = 0$ times/day and $Y_{\min} = 0$ minutes.

Programming the frequency and duration of the substrate's hydration

The hydration system can operate X times per day for a duration of Y minutes per hour. The maximum set values are $X_{\max} = 2$ times/day and $Y_{\max} = 15$ minutes while the minimum ones are $X_{\min} = 0$ times/day and $Y_{\min} = 0$ minutes

4.3.2. Measuring the temperature of the substrate

The temperature of the substrate was being measured at regular time intervals throughout the composting duration. For these measurements a microchip was used (DATA LOGGERS MODEL 100 2K). The device was installed in the interior of the bioreactor and was collected at the end of each composting trial and after the collection and removal of the end product. The recorded temperature values were obtained, using the software of the device. For comparison reasons and for the better understanding of the temperature profile during the composting processes, additional temperature measurements of the substrate have been performed using a portable thermometer. These temperature values were recorded once per day at the same point inside the bioreactor.

4.3.3. Measuring the moisture of the substrate

The moisture level was being recorded once per day for the duration of the composting process. Six same weighted and representative samples were being selected daily from inside the bioreactor. The final sample was formed after stirring and mixing the six samples together to form a homogeneous material. From the homogeneous material six separate samples were selected weighted and dried at 105°C for 12hrs. The dried samples were then placed inside a desiccator for cooling and weighting. The moisture content was evaluated from the average value of the six separate samples.

4.3.4. Measuring the oxygen content of the substrate

The oxygen level was being recorded once per day for the duration of the composting process. The measurements were being recorded using a portable oxygen meter (model OA 2) and setting the device at the same point inside the bioreactor.

4.3.5. Reprogramming the plc of the in-vessel bioreactor

The plc of the system was reprogrammed in case of suspending the in-vessel bioreactor operation due to potential malfunction. The reprogramming of the plc involved the adjustment of the agitation, hydration and aeration system settings that are performed by the PLC automatic control system according to the specifications stated in paragraph 4.3.1.

4.3.6. Monitoring and maintenance during composting

Throughout the duration of the composting process the electromechanical equipment and the rest of the bioreactor components and infrastructure were being monitored daily in order to maintain their operational status. The regular maintenance of all the electromechanical equipment of the system was performed according to the suggestions of the manufactures. In addition daily cleaning of the floor and surface of the bioreactor was performed in order to obtain high hygiene level for the personnel.

4.3.7. Disruption and start up of the bioreactor operation

Describes potential disruptions that had been occurred during the composting trials with respect to the operation of the in-vessel bioreactor.

4.3.8. Restoration of malfunctions and maintenance of the bioreactor

Involves the actions that had been initiated to restore potential malfunctions during the composting trials.

4.4. Sampling and analysis of the substrate

Six successive samples were being selected from inside the bioreactor in regular time intervals, usually every 5 to 7 days, throughout the composting process. The final sample was formed after stirring and mixing the six samples together to form a homogeneous material. From this homogeneous material six separate samples were selected from which a series of parameters were evaluated. The average value of each parameter was calculated from the average value of the separate samples. During each composting trial the following parameters were examined: pH, total carbon, total nitrogen, nitrates ($\text{NO}_3^- - \text{N}$) and ammonium nitrogen ($\text{NH}_4^+ - \text{N}$). Biological and micro-biological analyses have also been performed to examine the total and faecal coliforms and helminth eggs density to the sludges used in each trial.

4.5. Sampling and analysis of the substrate's leachate

Composting processes produce a natural liquid residue known as leachate which can be highly polluting. The leachate collection takes place via the removal portals at the bottom side of the bioreactor which acquire a $\frac{1}{2}$ inch pipe system for the continuous bioreactor leachate draining. On regular bases leachate samples were collected in order to be analysed throughout the duration of the composting trials. The examined parameters of the produced leachates for each composting trial were the pH, BOD_5 , nitrates ($\text{NO}_3^- - \text{N}$), ammonium nitrogen ($\text{NH}_4^+ - \text{N}$) and heavy metals concentration (Cd, Cr, Cu, Ni, Pb, Zn).

4.6. Removal and collection of the composting end product

The removal and collection of compost was performed at the end of each the composting trial through the removal portals that are situated at the bottom part of the bioreactor. To facilitate the discharge of the end product the agitation system was operated for small intervals. The compost was collected manually using shovels.

4.7. Sampling and analysis of the produced compost

At the end of each composting trial the produced end product was collected from the bottom layer of the in-vessel bioreactor. The collected compost was then mixed

sufficiently and spread outside the bioreactor's installation building to form a pile. Six representative samples were being collected from different points within the pile. The final sample was formed after stirring and mixing the six samples together to form a homogeneous material. From this homogeneous material six separate samples were selected from which a series of parameters were evaluated. The average value of each parameter was calculated from the average value of the separate samples. The examined parameters of the end product for each composting trial were the water content, pH, total carbon, total nitrogen, C:N ratio, nitrates ($\text{NO}_3^- - \text{N}$), ammonium nitrogen ($\text{NH}_4^+ - \text{N}$), total phosphorous as P_2O_5 , total potassium as K_2O , magnesium, manganese, calcium and heavy metals concentration (Cd, Cr, Cu, Ni, Pb, Zn). To estimate the reduction of the pathogenic microorganisms biological analyses have been performed to evaluate their density prior and after the composting processes. The analyses evaluated the density of the total and faecal coliforms and helminth eggs in sludges and in the compost produced from each trial.

4.8. Preparing the system for the implementation of the next composting trial

Monitoring and maintenance of the in-vessel bioreactor and the rest of the equipment and infrastructure was performed in order to prepare the system for the implementation of the next composting trial.

4.9. Assessment of the composting trial in order to improve the next trial

At this point a thorough evaluation was performed on the obtained results and observations during the composting trials as well as on the analysis of the problems which arose during the implementation of composting. The conclusions that were made were taken into consideration for the formation of the next composting trials.

5. Results and discussion

The composting trials that have been performed focus on the compost quality characterization and on the effects of unit operational conditions on the efficiency of the composting process, as well as on the end product quality. This section describes a very crucial part of the MOROCOMP project which is involved with the operation of different composting trials, the performance of the composting processes and the evaluation of the end product. The evaluation and the conclusions that have been made are based on the comparison of the experimental results with literature references on the operational and quality parameters that influence composting process and the quality of the end product.

5.1. Composting using primary sludge and green waste – 1st trial

In this section the experimental results obtained from the analysis of feedstock of the 1st composting trial are shown. The feedstock includes primary sludge from El Jadida city and sugar beet leaves.

5.1.1. Sampling and analysis of the raw material

The characterisation of the raw material that was used as a feedstock into the in-vessel composting bioreactor is essential for the evaluation of the aerobic composting processes and thus to the evaluation of the end products. The feedstock determines a priori the physical chemical and biological conditions involved in the composting processes. The rate at which materials compost and the quality of the final composted organic material are largely depended on the initial selection and mixing of raw materials. Table 2 presents the physicochemical characteristics of the primary sludge and the sugar beet leaves that were used as feedstock for the operation of the 1st composting trial.

Table 2: The physicochemical characteristics of the raw material used in the 1st composting trial

Parameter	Primary sludge	Sugar beet leaves
Dry Solids (% d.s.)	27.8	51.65
pH	6.8	7.1

Total C (% d.s)	10.5	59.94
Total N (% d.s)	1.76	3.52
C:N ratio	5.97	17.03
Total P, as P ₂ O ₅ (% d.s)	1.300	0.240
K, as K ₂ O (% d.s)	0.691	3.524
Ca as CaO (% of d.s)	4.873	3.387
Mg as MgO (% of d.s)	2.339	1.381
Cd (mg/Kg d.s)	0.986	0.113
Cr (mg/Kg d.s)	18.410	0.828
Cu (mg/Kg d.s)	141.471	7.924
Ni (mg/Kg d.s)	24.305	0.895
Pb (mg/Kg d.s)	81.094	0.504
Zn (mg/Kg d.s)	237.894	48.916

According to Table 2 the primary sludge acquired increased moisture content of 72.2% since the collected sludge did not undergo dewatering. On the other hand sugar beet leaves had much lower water content of 48.35%. Sugar beet leaves can balance the water level of the substrate when mixed with primary sludge. The pH of primary sludge and sugar beet leaves was near neutral which is considered satisfactory for the development of composting processes. With respect to the total carbon content sugar beet leaves acquired high carbon content (59.94%) while in primary sludge the total carbon was much lesser (10.5%). The nitrogen content was high proportionally to the carbon content, leading to low carbon to nitrogen ratios of 5.97 and 17.03 for the primary sludge and sugar beet leaves respectively. Furthermore, the physical properties of green waste make a good quality structuring agent for mixing with sewage sludge. Thus, appropriate blending of these materials with the examined sludge will have C:N and moisture values suitable for the development of microorganisms involved in composting.

The quantity of nutrients such as nitrogen (N), phosphorous (P), and potassium (K) incorporated in the raw material, influences the microbial activity during composting since these nutrients are involved with the protein synthesis of the microorganisms to build cell walls and other structures. In addition N, P and K are used in the greatest quantities by plants and are the nutrients most often applied through commercial

fertilisers. According to Table 2 the NPK value as well as the rest macro-elements, Ca and Mg, of the raw material considered to be sufficient for the purposes of composting.

With respect to the heavy metal content primary sludge did not have limiting heavy metal values for the composting process since it did not exceed the limits for sludge application in agricultural land as has been set by Directive 86/278/EEC⁴ (Table 3). Urban sewerage systems rarely transport only domestic sewage to treatment plants; industrial effluents and storm-water runoff from roads and other paved areas are frequently discharged into sewers. Thus sewage sludge often contains, in addition to organic waste material, traces of many pollutants such as heavy metals that are used in our modern society. Although urban residues are generally emphasized by high heavy metal content in the case of Moroccan sludge the concentration appeared to be low in comparison to the limit values for heavy metal concentrations in sludge for use in agriculture as shown in Table 3.

Table 3: Limit Values for Heavy Metal Concentrations in Sludge for use in Agriculture [11]

Parameters	Limit Values (mg/kg of dry matter)
Cadmium [Cd]	20 - 40
Copper [Cu]	1000 - 1750
Nickel [Ni]	300 - 400
Lead [Pb]	750 - 1200
Zinc [Zn]	2500 - 4000
Mercury [Hg]	16 - 25
Chromium [Cr]	-

The usual types of pathogens introduced in wastewaters and consequently in sludge consist of bacteria, viruses, protozoa, nematodes and fungi. These attack the human immune system causing diseases of the gastrointestinal tract such as typhoid, paratyphoid fever, dysentery, diarrhoea and cholera [1]. Table 4 shows that the primary sludge used in the 1st composting trial contains high number of pathogenic microorganisms which constitute a health hazard and if sludge is used for agricultural purposes without following a proper treatment process, such as composting, there is a strong risk of contamination [12, 13, 14, 15].

⁴ Directive 86/278/EEC on the protection of the environment and in particular of the soil when sewage sludge is used in agriculture

Table 4: Micro-biological parameters of primary sludge used in the 1st composting trial

Micro-biological parameters	Primary sludge
Total coliforms Log10 MPN/10g DS	9.53
Faecal coliforms Log10 MPN/10g DS	8.83
Helminth eggs /10g DS	23.00

5.1.2. Composition of the composting raw material

The feedstock material of the 1st composting trial comprised of primary sludge from El Jadida city and sugar beet leaves. The quantities of each material used are given in Table 5.

Table 5: Composition of the mix used for composting in the 1st composting trial

Raw material	Kg w.w.
Primary sludge	700
Sugar beet leaves	650

As has been stated previously primary sludge acquired high moisture content due to the fact that there was not dewatering installations in the region where primary sludge was produced. Therefore composting primary sludge individually was not considered appropriate and the addition of a bulking agent such as sugar beet leaves would modify the physical (moisture content, structural support, porosity, aeration) as well as the chemical properties (carbon to nitrogen ratio, nutrients content etc.) of the initial mixture in such manner as to the promote microbiological activity.

5.1.3. Pretreatment and preparation of the composting raw material

The sugar beet leaves were shredded to size 2 to 4 cm using a commercial shredder type MTD 118E prior its supply into the bioreactor. After the green material had been shredded it was then weighted and packed ready to be used. The primary sludge was also weighted and packed ready to be used. The weighted and packed raw materials were then mixed manually using shovels and ready to be fed into the bioreactor.

5.1.4. Feeding the in-vessel bioreactor

The feeding of the bioreactor was performed using the mixture prepared as indicated in paragraph 5.1.3 via the conveyor belt in which the feedstock was loaded manually using shovels.

5.1.5. Composting process control

Composting process control involves all the actions that had been planned and carried out with respect to the operational conditions of the in-vessel bioreactor.

5.1.5.1. Programming the plc of the in-vessel bioreactor

The operation of the agitation, hydration and aeration systems was performed by the PLC automatic control system. These operations can also be performed manually. The programming of each operation during the 1st composting trial is given below:

Programming the frequency and duration of the substrate's agitation:

The motor was set to operate 3 times per day (every 8 hours) for 5 minutes each time for the agitation of the substrate.

Programming the frequency and duration of the substrate's aeration:

The fan was programmed to operate 3 times per day (every 8 hours) for 5 minutes each time for the aeration of the substrate.

Programming the frequency and duration of the substrate's hydration:

The hydration system operated manually during the 1st composting trial although it could be programmed to operate automatically. The substrate was subjected to hydration whenever it was considered appropriate according to its moisture content that had been recorded from the daily measurements. The duration of the hydration operation as well as the water flow were also adjusted according to the moisture content of the substrate that had been recorded from the daily measurements

5.1.5.2. Measuring the temperature of the substrate

In the process of composting, microorganisms break down organic matter and produce carbon dioxide, water, heat, and humus, the relatively stable organic end product. Under optimal conditions, composting proceeds through three phases which may have considerable overlap based on temperature gradients and differential temperature effects on microorganisms. These are 1) the mesophilic, or moderate-temperature phase, which lasts for a couple of days, 2) the thermophilic, or high-temperature phase, which can last from a few days to several weeks, and finally, 3) the cooling and maturation phase which results to the stabilisation of compost. Figure 2 presents the temperature evolution during the 1st composting trial. The temperature values are the mean values obtained from the temperature data loggers and the temperature probe.

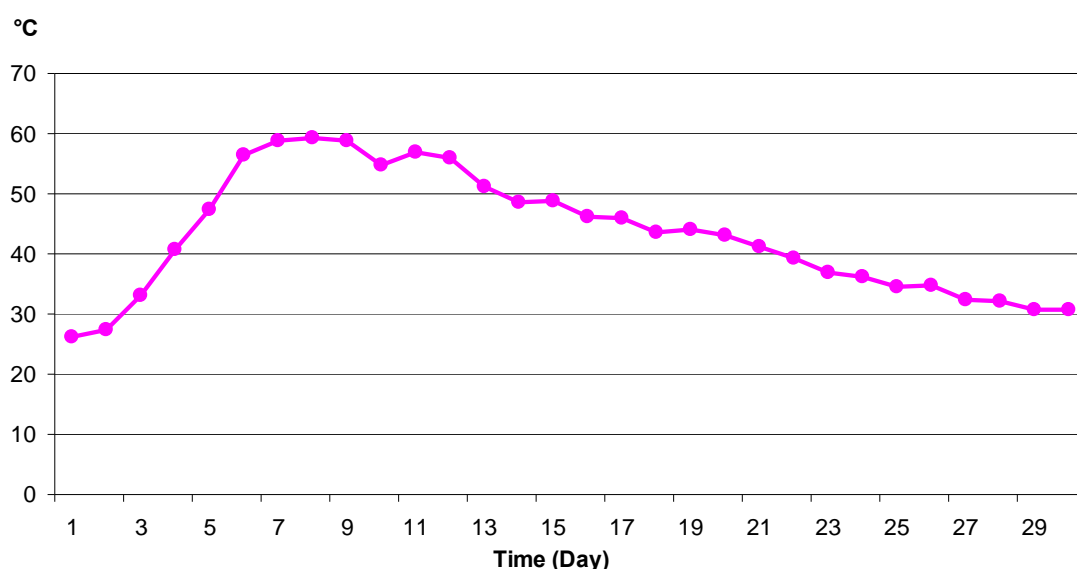


Figure 2: Temperature profile during the 1st composting trial

Temperature measurements during the 1st composting trial showed that the substrate passed from an initial mesophylic phase (<40°C) to a thermophylic stage approximately on the 4th day. The initial decomposition of the organic matter is carried out by mesophilic microorganisms, which rapidly break down the soluble, readily degradable compounds which are present in the primary sludge compounds. Microorganism activity releases large amounts of energy in the form of heat. This heat accumulates due to the self-insulating qualities of the compost material and causes the temperature to rise rapidly. As the temperature was rising above about 40°C, the mesophilic microorganisms become less competitive and were being

replaced by others that are thermophilic, or heat-loving. Compared with the initial mesophilic phase, the degradation rates were higher during the thermophilic phase as it is shown by the elevated temperatures and the increased microbial activity compared to the mesophilic phase. During the thermophilic phase the maximum temperature was achieved on the 8th day which was approximately 60°C while elevated temperatures ranging from 55°C to 60°C remained in the mixture for 7 continuous days (6th to 12th day) which is sufficient time for the sterilisation of the substrate from potential pathogenic microorganisms. Only after all substrate, capable of promoting microbial activity, had been used up did the temperature begin to fall to reach a second mesophylic phase while the maturation process took place during this last stage of the process.

5.1.5.3. Measuring the moisture of the substrate

The moisture content of compost varies depending on the porosity of the reactor feed, free air space, aeration-oxygen, temperature, and other related physical factors. Moisture content (%) is defined as weight loss after the sample has been dried to constant weight at 105°C for 12hrs. Moisture is essential for all living organisms since it supports their metabolic processes while water is the medium for the chemical reactions, transportation of nutrients and allows the microorganisms to move about. Most microorganisms are very sensitive to this factor in their environment. When the moisture content of an actively composting mixture falls between 35% and 40% of the total weight, decomposition rates decelerate significantly as microbes are less able to carry out their metabolic activities; below 30% they essentially stop. On the other hand, moisture level higher than 60% can quickly lead to anaerobic conditions and foul-smelling as water fills the pore spaces of the organic mixture impeding oxygen diffusion through the composting material. This leaves no room for air, a condition that is not favorable for aerobic microorganisms. For most compost mixtures, 55% to 60% is the recommended upper limit for moisture content while the optimum value is between 40 to 65% of the total weight of the composting material. Figure 3 presents the moisture evolution during the 1st composting trial.

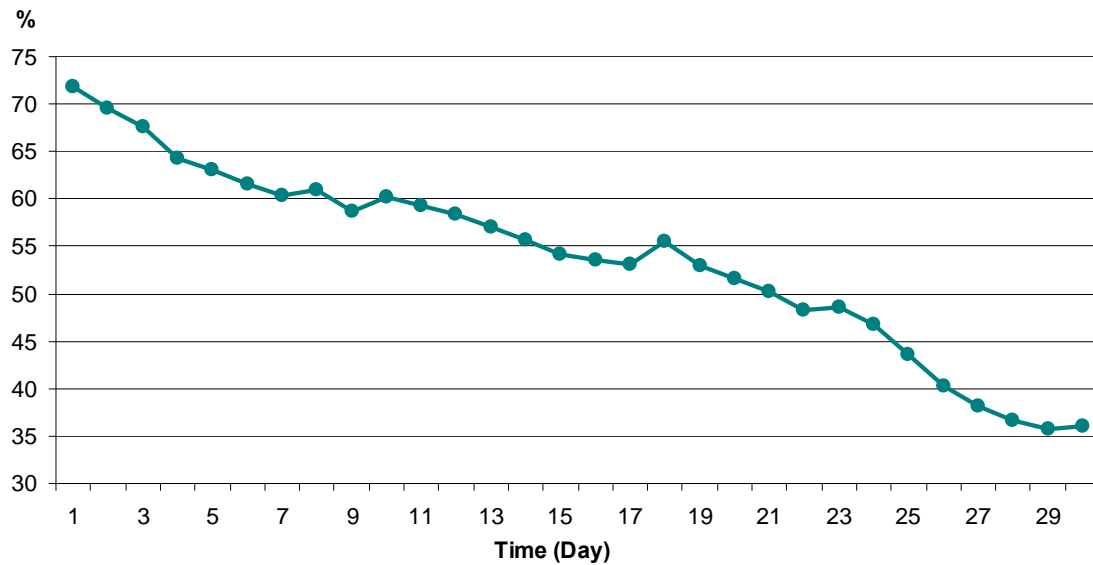


Figure 3: Moisture content profile during the 1st composting trial

From the results obtained for the 1st composting trial, the initial substrate appeared to have increased moisture content (71.8%) which was steadily reduced due to the initial decomposition of the readily degradable organic matter carried out by mesophilic microorganisms. After the 8th day of the process the moisture content had fallen less than 60%. This level of moisture content in conjunction to sufficient agitation and oxygen supply of the substrate enhanced the metabolic activities of microbes and led rapidly to higher temperature values. Elevated temperature values (thermophilic phase) reduced even more the moisture content due to water evaporation which at the end of the composting process, on the 30th day, was at 36.1%. Low water level reduces the intensive microbiological activity. Also at the same time reduces the transportation costs of compost, so it is required to have low water content when the compost is mature. It is worth mentioning that spontaneous water content increases were recorded during the composting process on the 8th, 10th, 18th and 23rd day due to the hydration of the substrate as stated in paragraph 5.1.5.1 in order to maintain moisture at desirable levels.

5.1.5.4. Measuring the oxygen content of the substrate

The in-vessel composting system was designed to operate in aerobic conditions during which the decomposition of the organic matter takes place in the presence of oxygen. Oxygen is an essential ingredient for successful composting since microorganisms oxidize carbon for energy emitting carbon dioxide. Therefore the

ventilation of the substrate is a very basic parameter of the composting process since it concerns bio-oxidation processes of the organic substrate. Aeration has multiple functions (i) it provides sufficient oxygen to the compost mixture, (ii) it controls the systems temperature and (iii) it removes moisture as well as CO₂ and other gases resulting in a more efficient compost process. The air supply contributes significantly to lowering the substrate's moisture, since the moisture of sludge is high. Moisture content, temperature and aeration (oxygen supply) of compost are linked together since microbial activity and thus elevated temperatures require a certain level of moisture content without the compost material being too wet and heavy so that free air space will be sufficient between the particles for air to diffuse into it. Although composting process takes place even in low oxygen content (5%), oxygen concentrations greater than 10% are considered optimal for maintaining aerobic conditions and thus sustaining aerobic microbial activity [16]. Figure 4 presents the oxygen evolution during the 1st composting trial.

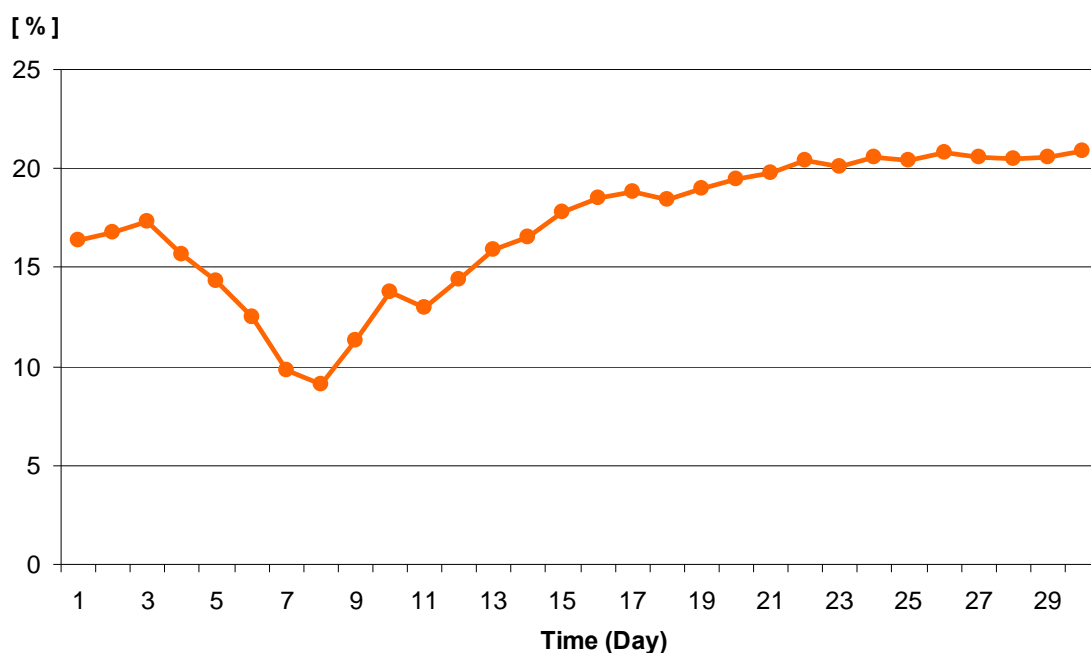


Figure 4: Oxygen profile during the 1st composting trial

According to Figure 4 the oxygen concentration of the substrate was maintained greater than 10% for the majority of the 1st composting trial duration. Furthermore an incremental tendency of oxygen concentration was achieved throughout the process indicating that the agitation and aeration of the substrate was sufficient in promoting aerobic conditions in the bioreactor. The lowest oxygen values just below 10% were observed during the thermophilic phase of composting due to the high oxygen

demand for the oxidation of the available organic matter. In addition, at the latest stage of the process, the level of oxygen increased and exceeded the 20% on the 22nd day indicative of the low rate of organic matter decomposition since the oxygen nearly reached the atmospheric oxygen level. It must also be mentioned that the steep decrease of oxygen content on the 8th day of the process was due to the suspension of the agitation system operation which was caused by a malfunction to the bioreactor's motor which was restored immediately as explained below.

5.1.5.5. Reprogramming the plc of the in-vessel bioreactor

The agitation system operation was out of order on the 8th day of the composting process since a malfunction occurred to the bioreactor's motor and all the operations of the automated control system were suspended. At that time the aeration of the substrate was handled manually from the control panel. After the problem had been restored the operation of the bioreactor was reprogrammed and restarted using the initial settings stated in paragraph 5.1.5.1.

5.1.5.6. Monitoring and maintenance during composting

Throughout the duration of the composting process the electromechanical equipment and the rest of the bioreactor's components and infrastructure were monitored daily in order to maintain their operational status. The regular maintenance of all the electromechanical equipment of the system was performed according to the suggestions of the manufacturer (e.g. lubrication, cleaning the filter of the ventilation system, cleaning the head of the piping system, leachate removal). In addition, daily cleaning of the floor and surface of the bioreactor was performed in order to obtain high hygiene level for the personnel.

5.1.5.7. Disruption and startup of of the bioreactor operation

The disruption of the in-vessel bioreactor operation on the 8th day involved only the operation of the agitation system of the bioreactor and that of the hydration system. The operation of the ventilation system was controlled so that the gases emitted during the process were being removed through the bioreactor's air suction system

which disperses gases through an air-pipe to the bio-filter area⁵ in order to prevent undesirable odours within the building where the bioreactor is installed.

5.1.5.8. Restoration of malfunctions and maintenance of the bioreactor

During the 8th day of the composting process a malfunction occurred to the motor of the in-vessel bioreactor. The chain providing motion to the revolving axle was broken. The revolving axle comprises of stirring elements responsible for the agitation of the substrate. The malfunction was restored and the bioreactor started to operate again on the 9th day of the composting process.

5.1.6. Sampling and analysis of the substrate

Substrate samples were taken every 6 to 8 days of the composting process and the examined parameters included the pH, total carbon, total nitrogen, nitrates ($\text{NO}_3^- - \text{N}$) and ammonium ($\text{NH}_4^+ - \text{N}$).

5.1.6.1. pH

The acidity or alkalinity of the material, referred to as pH, is also an important parameter of composting. The pH determines the species of microorganisms that will be developed for the biodegradation of the organic fractions. The effects of pH on the composting process are directly related to the effect of pH on microbial activity and more specifically on microbial enzymes. However composting process is relatively insensitive to pH within the range commonly found in mixtures of organic materials. Although composting process can work over a wide spectrum of pH values, a range between 6.0 and 8.5 is preferred [2, 3]. Figure 5 presents the pH evolution during the 1st composting trial.

⁵ the bio-filter bed was filled with the end product resulted from the 1st composting trial

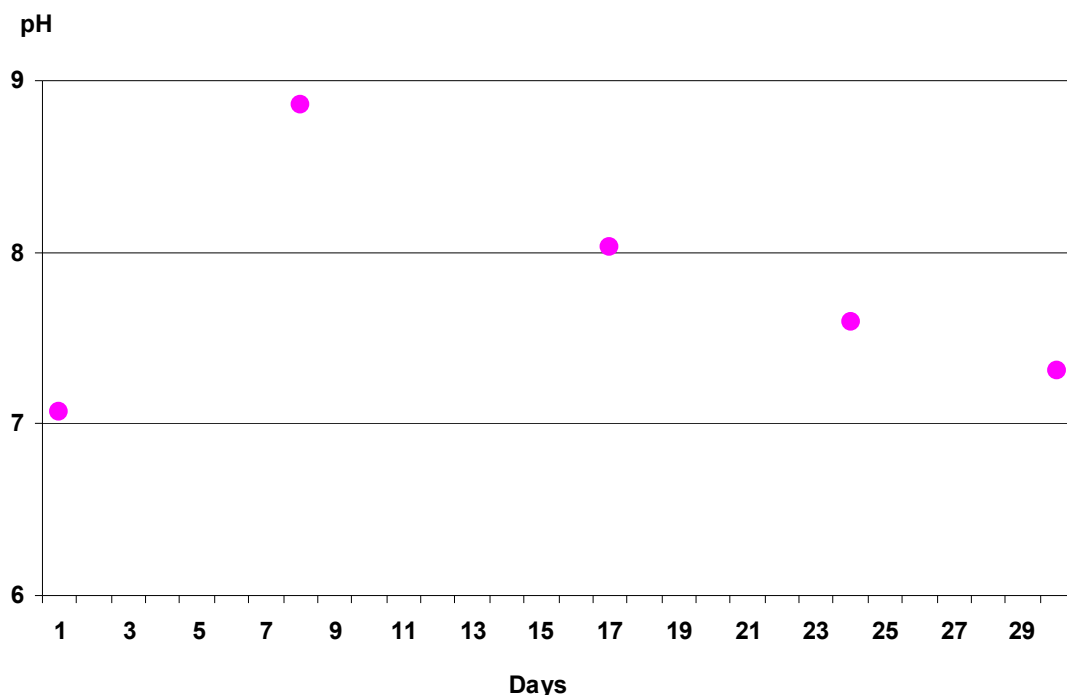


Figure 5: Changes in pH values during the 1st composting trial

The pH of the extracts increased during the first 8 days of the 1st composting trial. This is more probably related to the degradation of the organic matter, which releases ammonia [17]. At the onset of composting, compounds such as amino acids, proteins etc. were mineralized through the activity of proteolytic bacteria to give ammonia and thus the pH value increased reaching a maximum of 8.86 on the 8th day. Thereafter, through ammonia volatilization and its oxidation to nitrates by the action of nitrobacteria, the ammonia content decreased and thus the pH value drops from 8.86 to 7.31 on the 30th and final day of the process [12, 18].

5.1.6.2. Total Carbon

During composting, controlled microbial transformation of the organic matter that is present in the compostable material takes place. Part of the organic matter is mineralized to carbon dioxide, ammonia and water. The carbon loss occurs through bio-oxidation of organic carbon to CO₂ during composting. The CO₂ can escape as a gas or dissolve in the liquid, forming carbonic acid (H₂CO₃) bicarbonate (HCO₃⁻) and carbonate (CO₃²⁻). The other part of the organic carbon is transformed into humic substances which are structurally very similar to those present in soils. Figure 6 presents the total carbon evolution during the 1st composting trial.

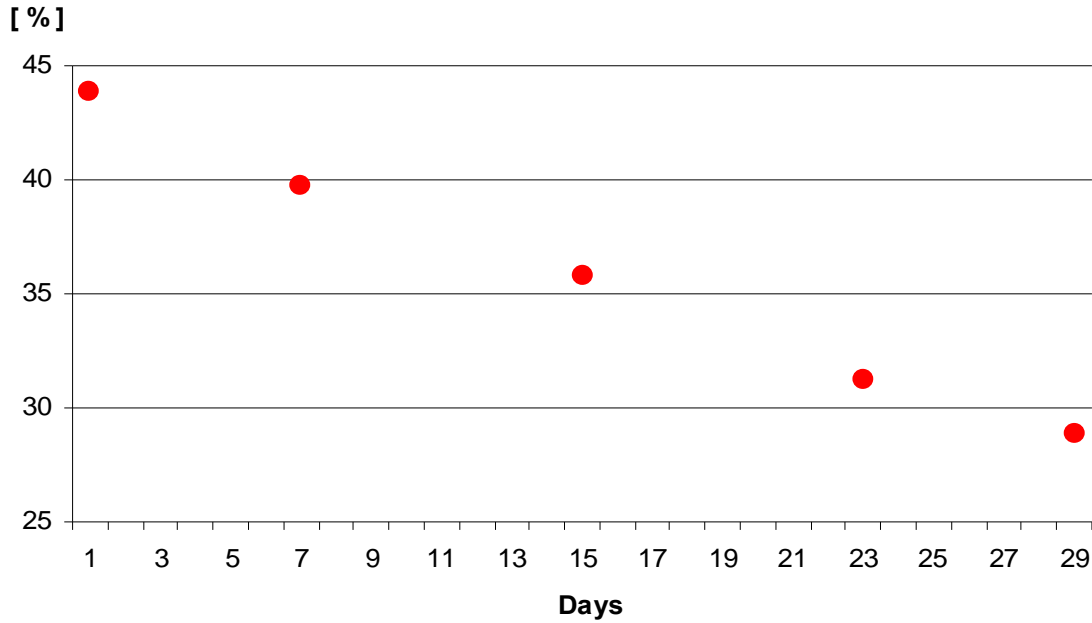


Figure 6: Changes in total carbon content [%] during the 1st composting trial

The total carbon content of the substrate presents a linear inclination throughout the 1st composting trial duration. The total carbon content of the substrate at the initial stage of composting (1st day) was 43.87% which was subjected to a 15.03% reduction throughout the process mainly due to the microbial attack on the most labile organic substances and their disappearance through mineralization. At the end of the process on the 30th day the total carbon content of compost was 28.84% which is considered as of good quality in regard with its carbon content [19].

5.1.6.3. Total Nitrogen

Nitrogen is a crucial component of proteins, nucleic acids, amino acids, enzymes and co-enzymes needed for microbial cell growth. During composting the nitrogen is metabolized mainly to ammonium while the non soluble complexes of nitrogen decompose to soluble nitrogen forms that are readily available for metabolic activities [20]. Gaseous nitrogen losses during composting occur mainly as ammonia but may also occur as nitrogen and nitrates oxides [21]. Witter and Lopez-Real [22] reported that total nitrogen loss could amount to 50% of the initial nitrogen in composting of sewage sludge and straw mixtures while Hansen et al. [23] stated that nitrogen losses increased up to 33% during composting of poultry manure. Figure 7 presents the total nitrogen evolution during the 1st composting trial.

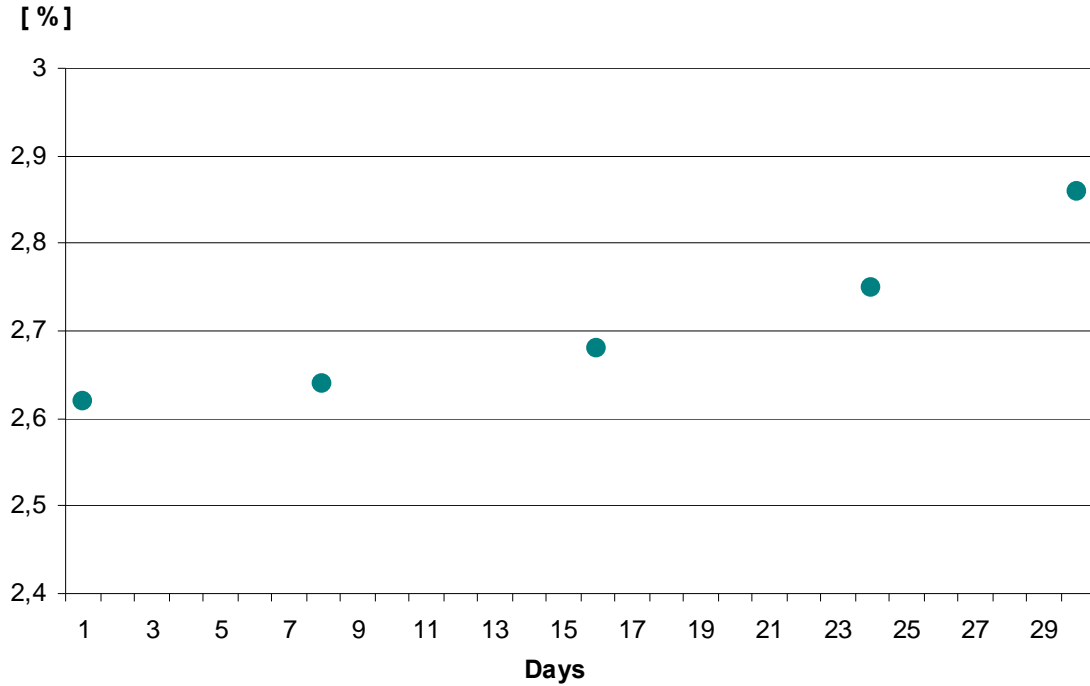


Figure 7: Changes in total nitrogen content [%] during the 1st composting trial

Due to the activity of proteolytic bacteria, which is initiated when substrate's temperature rises at the initial stage of composting, ammonium is produced. A proportion of the available nitrogen is volatilized and escape in the atmosphere as ammonia [24]. These losses are greater at elevated temperatures and pH values and when the carbon to nitrogen ratio (C:N) is low but also agitation and aeration rate may affect the rate of ammonium volatilization [24]. Since high temperatures are fundamental in aerobic composting and destruction of pathogen, not much can be done about controlling temperatures other than to avoid temperatures above 70°C which retard bacterial activity and permit ammonium accumulation. The activity of proteolytic bacteria is reduced rather shortly while the activity of nitrogen-fixing bacteria even at limited scale enriches the nitrogen content of compost especially during the latest stages of composting [20, 25, 26]. Therefore the total nitrogen content decreases during the process of composting mainly through ammonia volatilization. This nitrogen loss can be evaluated in terms of absolute value. On the other hand, in terms of dry weight, there is an increase in total nitrogen concentration due to the mineralization of organic matter and consequent loss of weight in the mass being composted through losses of CO₂ and H₂O [3]. Increase of total nitrogen content has been observed in composting processes in which the organic matter has been reduced significantly [27]. Other studies confirm that additional increase may be

acquired due to processes of non-symbiotic nitrogen fixation which occur due to the activity of azotobacters which recover a proportion of nitrogen, mainly at the latest stage of the composting, which has been lost as ammonia [2, 28]. The nitrogen content at the end of the 1st composting trial on the 30th day was 2.86% which is higher than the initial nitrogen content (2.62%).

5.1.6.4. Ammonium Nitrogen ($\text{NH}_4^+ - \text{N}$)

As has been mentioned earlier, during composting the nitrogen is metabolized to ammonium due to the activity of proteolytic bacteria. Figure 8 presents the ammonium evolution during the 1st composting trial.

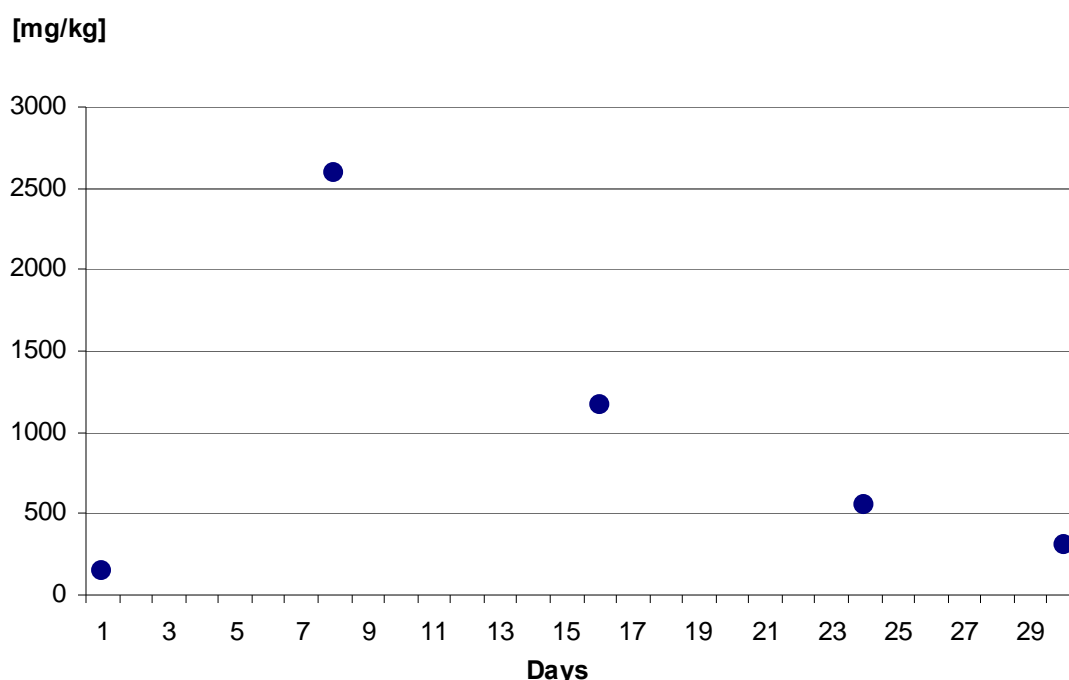


Figure 8: Changes in ammonium concentration [mg/kg] during the 1st composting trial

According to Figures 2 and 8 the conversion rate of nitrogen to ammonium was increasing as the temperature values of the substrate increased [29]. The highest ammonium conversion occurred during the early stage of active decomposition in which elevated temperature values of the substrate (up to 59.2°C on the 8th day) enhanced the formation of ammonium reaching its maximum value of 2594.25mg/kg on the 8th day of composting. From that point onwards the ammonium concentration was reduced significantly mainly due to the stimulation of nitrification and reached 302.62mg/kg at the final day of composting. However, the rapid decrease in

ammonium during composting did not coincide with a rapid increase in nitrates concentration shown in Figure 9. The concentration of nitrates was very low initially suggesting that nitrogen was lost during composting. Losses of nitrogen in this composting process were governed mainly by volatilization of ammonia due to high pH and temperatures values of the substrate. Agitation and aeration rate may have also affected the rate of ammonia volatilization [30].

5.1.6.5. Nitrates (NO_3^- -N)

As has been observed by recent studies, during composting the ammonium content is reduced due to the stimulation of nitrification while the concentration of nitrates is increased [31, 32, 33]. The conversion of ammonia to nitrates is due to the activity of nitrifying bacteria as shown by the following reactions.



There are two different types of nitrification that occur during composting. The first type is the heterotrophic nitrification which takes place at the initial phase of composting. The conversion of ammonia to nitrates at the initial stage of composting is exclusively due to the activity of heterotrophic nitrifiers [25, 34]. On the contrary autotrophic nitrification is developed at a later stage of composting since biooxidation of ammonium to nitrates during autotrophic nitrification is restricted by elevated temperatures and by increased ammonium concentrations of the compostable matter [35, 36]. Figure 9 presents the nitrates evolution during the 1st composting trial.

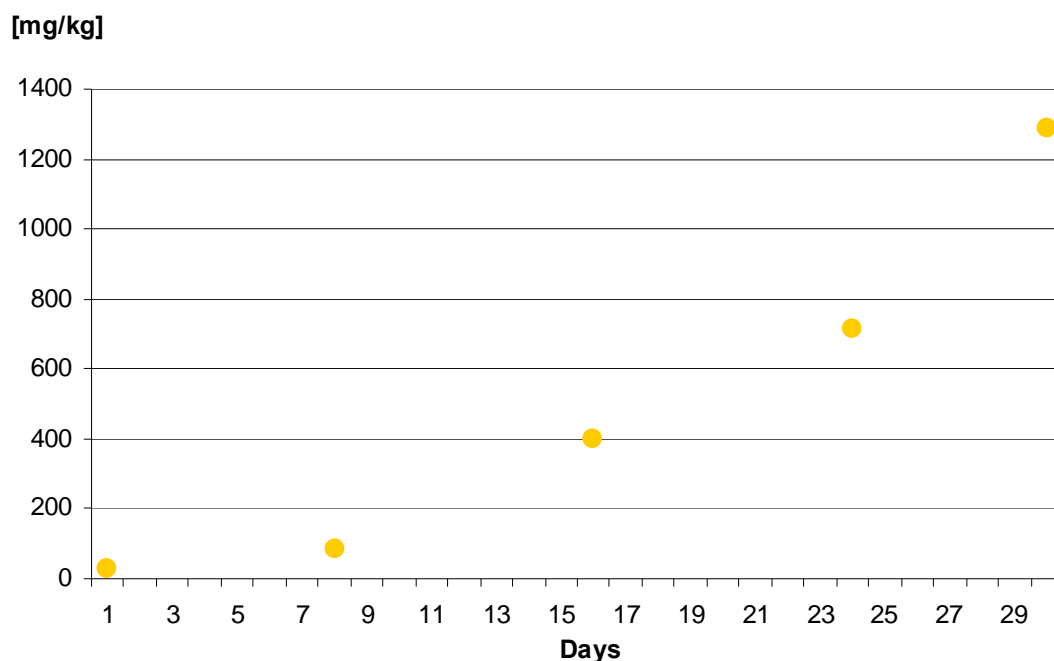


Figure 9: Changes in nitrates concentration during the 1st composting trial

According to Figure 9 during the first days of the 1st composting trial the nitrates content increased slowly (26.84 mg/kg to 82.31 mg/kg on the 1st and 8th day respectively) due to heterotrophic nitrification. However, as composting was developing nitrates concentration presented a significant increase which can be explained by the activity of autotrophic nitrobacteria which oxidize ammonium compounds into nitrates in the presence of oxygen-rich environment, as has been shown in Figure 4. Nitrates reached at 1286.48 mg/kg on the 30th and final day of the process.

5.1.6.6. C:N Ratio

In the composting process it is essential to determine the nutrient content of the feedstock. Of the many elements required for microbial decomposition, carbon and nitrogen (C:N ratio) are the most important. Carbon provides both an energy source and the basic microbial building block since it constitutes approximately half of the mass of microbial cells. Nitrogen is a crucial component of proteins, nucleic acids, amino acids, enzymes and co-enzymes needed for microbial cell growth.

According to various studies on municipal solid waste composting the optimum C:N ratio for composting processes ranges from 25:1 to 35:1, that is 25 to 35 parts of

carbon for each part of nitrogen by weight, although ratios from 20:1 up to 40:1 can give good composting results [24, 25, 37, 38]. Low ratios supply the mixture with excess of nitrogen which is lost as ammonia gas⁶ creating undesirable odors whereas high C:N ratios cause shortage of nitrogen and the degradation rate of the composting material remains relatively low since micro-organisms must go through many life-cycles, oxidizing off the excess carbon until a more convenient C:N ratio for their metabolism is reached. In addition nitrogen limitation may lead to extensive organic acid formation from carbonaceous waste, which tends to lower the pH and thereby retard the microbial activity. The ratio is weighted in favor of carbon, because uses for carbon outnumber those for nitrogen in microbial metabolism and synthesis of cellular materials. Not only is carbon utilized in cell wall or membrane formation, protoplasm, and storage products synthesis but also an appreciable amount is also oxidized to CO₂ in metabolic activities. On the other hand, nitrogen has only one major use as a nutrient namely as an essential constituent of protoplasm. Consequently, much more carbon than nitrogen is required. Figure 10 presents the carbon nitrogen ratio evolution during the 1st composting trial.

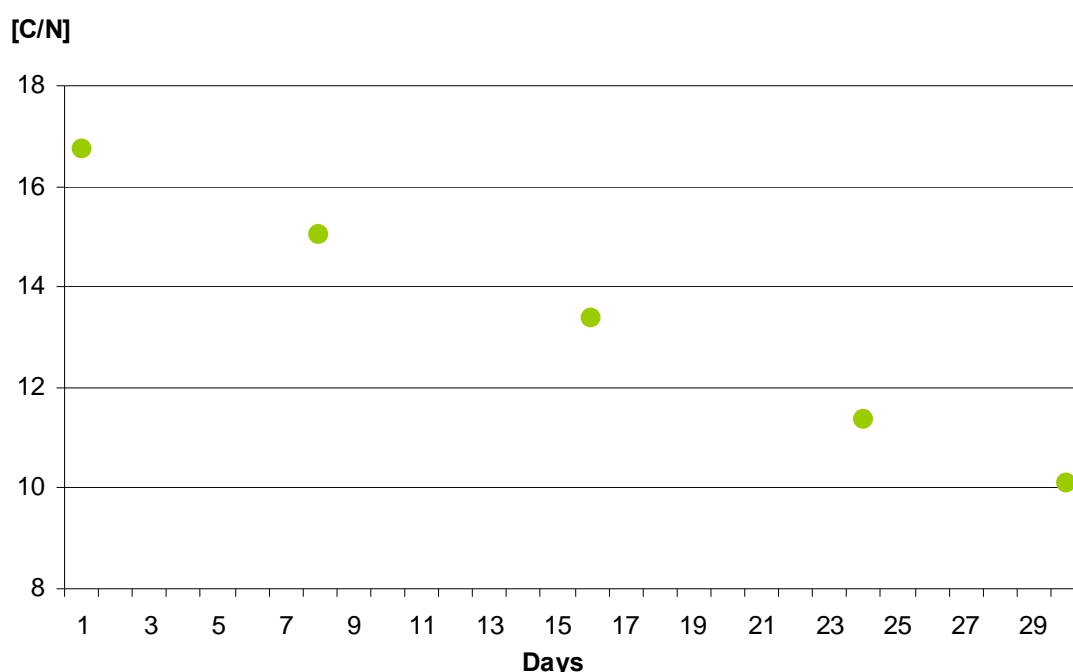


Figure 10: Changes in C:N ratio during the 1st composting trial

According to Figure 10 during the 1st composting trial, the C:N ratio presents a steadily downwards inclination. This inclination can be explained by the carbon loss

⁶ leads to excess ammonia formation, which increases the pH and thereby enhances ammonia volatilization

which occurs through the bio-oxidation of organic carbon to CO₂, shown in Figure 6. The initial substrate acquired a 16.74 C:N ratio which is lower than the optimal range of carbon to nitrogen ratios since primary sludge and the sugar beet leaves that were used as supplement had a low C:N ratio as shown in Table 2. At the end of the process on the 30th day the C:N ratio has been decreased to 10.08. With respect to the C:N ratio the finished compost was qualified as of good quality compost which can be applied in agricultural land. This is so due to the fact that composts acquiring a C:N ratio higher than 20 interfere with plant nutrition because soil microflora competes with the plants roots for nitrogen [2, 15].

5.1.7. Sampling and analysis of the substrate's leachate

Leachate can be used as an indicator to evaluate composting processes. Leachate is produced from moisture extraction of the substrate mainly due to its hydration and partly by the molecular water resulting from the biooxidation of the organic matter. Table 6 presents the evolution of the leachates parameters examined during the 1st composting trial.

Table 6: Characteristics of leachates resulting from the 1st composting trial

Parameter/Time	3rd day	7th day	15th day	23rd day
pH	8.0	8.7	7.9	7.5
BOD ₅ (mg/L)	50984	31015	17972	8958
NH ⁴⁺ - N (mg/L)	823.47	1985.06	895.10	450.84
NO ³⁻ - N (mg/L)	34.05	55.74	338.73	687.51
Cd (mg/L)	0.00138	0.00829	0.00041	0.00034
Cr (mg/L)	0.01154	0.00903	0.08225	0.00716
Cu (mg/L)	0.14307	0.13555	0.14676	0.15023
Ni (mg/L)	0.02818	0.02316	0.02185	0.01739
Pb (mg/L)	0.03655	0.03782	0.03466	0.02684
Zn (mg/L)	0.21343	0.19946	0.23631	0.26031

The pH values of leachates change in the same manner as the pH values of the substrate since the physicochemical conditions involved in composting are the same in the liquid and in the solid state. The pH in leachates increased during the initial stage reaching to a maximum of 8.7 and then it was gradually reduced acquiring a near neutral pH of 7.5 at the end of the process.

Biochemical Oxygen Demand (BOD₅) is a measure of the amount of oxygen that bacteria will consume while decomposing organic matter under aerobic conditions. Biochemical oxygen demand is determined by incubating a sealed sample of water for five days and measuring the loss of oxygen from the beginning to the end of the test [39]. According to Trujillo et al. leachates originating from wastewater sludge composting processes are characterized by increased BOD₅ values reaching up to 70 g/L [40]. The initial sample of leachates derived from the 1st composting trial acquired an increased initial BOD₅ value of 50.9g/L on the 3rd day. As the process develops the organic intermediate products undergo complete biooxidation to CO₂ while the non biodegradable or less susceptible to degradation compounds accumulate. Therefore the microbial activity gradually decreases and the used oxygen by the biological organisms in leachates is reduced as the process continuous. For this reason the BOD₅ evolution during composting presented a downwards inclination and at the end of the process leachates had been reduced approximately 1/6 of the initial BOD₅ value. Leachates acquired at the initial stage of composting a light dark colour which was gradually becoming darker as composting was developing. This is indicative of the presents of humic/fulvic and other complex compounds which are dark coloured, hydrophilic organic compounds whose molecular weight varies from few hundreds to few thousands. Ammonium and nitrates in leachates followed the same pattern as the ammonium and nitrates of the substrate indicating that the composting process was well monitored since the conditions were in favor of nitrification. Ammonium ions concentration presented a steep increase during the first days of composting reaching to a maximum of 1985.06mg/L on the 7th day. From that day onwards ammonium concentration kept reduced and on 23rd day their concentration in leachates was 450.84mg/L. Nitrates in leachates presented a significant increase throughout the duration of the 1st trial. On the 3rd day their concentration was limited at 34.05mg/L while on the 23rd day it had been increased approximately 12 times reaching at 687.51mg/L. Table 6 shows also that heavy metals such as Cd, Cr, Cu, Ni, Pb and Zn were present in leachates throughout the process. The metal concentrations were much below the indicative values for wastewater disposal as shown in Table 7.

Table 7: Suggested limit values for wastewater application in water recipients based [41]

Parameters	Sewer	Rivers	Sea
BOD ₅ (mg/L)	500	40	40
COD	1000	120	150
Temperature (°C)	35	28	35
pH	6-9	6-9	6-9
Suspended Solids (mg/L)	500	50	40
SO ₄	4.0	1.0	0.6
NO ₃	20	4	20
NH ₄	25	10	20
SO ₄	1500	100	-
Phenols	0.01	0.01	0.005
Al (mg/L)	10	1	5
As (mg/L)	0.5	0.1	0.5
Cd (mg/L)	0.5	0.1	0.5
Mn (mg/L)	3	0.1	0.5
Pb (mg/L)	10	2	-
Ni (mg/L)	5	0.5	0.1
Fe (mg/L)	10	0.5	2.0
Hg (mg/L)	15	2	2
Cr ⁺³ (mg/L)	2	1	2
Cr ⁺⁶ (mg/L)	0.5	0.2	0.2

According to the BOD₅, ammonium and nitrates values obtained for the leachates of the 1st composting trial it can be concluded that leachates had a heavily polluted organic and inorganic load which needs to be treated before its final disposal. The quantity of leachate production during the 1st trial was limited and stored to an impermeable reservoir for sufficient period of time to be stabilized at lower values through aeration. Although composting can be assessed for sludge treatment and reuse through land application, leachates remain an environmental problem since the treatment of solid waste through composting results in transferring the problem from solid to liquid phase. Leachates must be treated accordingly in order to be disposed to a water recipient based on the suggested standards shown in Table 7. However

Directive 91/271/EEC⁷ seeks to harmonise measures relating to the treatment of wastewater at Community level in order to protect the environment from any adverse effects due to discharge of such waters [42].

5.1.8. Removal and collection of the composting end product

The removal and collection of compost was performed on the 30th day of the composting process through the removal portals that are situated at the bottom part of the bioreactor. To facilitate the discharge of the end product the agitation system was operated for small intervals. The compost was collected manually using shovels and it was used as filling material to the biofilter bed.

5.1.9. Sampling and analysis of the produced compost

In order to evaluate the quality of the end product resulting from the 1st composting trial a series of parameters were examined. Table 8 presents the physicochemical properties of the compost produced from the raw material shown in Table 5 which acquire the characteristics presented in Table 2.

Table 8: Physicochemical parameters of the compost produced in the 1st composting trial

Parameter	Compost
Dry Solids (% d.s)	63.90
pH	7.31
Total Carbon (% d.s)	28.84
Total Nitrogen (% d.s)	2.86
C:N Ratio	10.08
NO ₃ ⁻ - N (mg/Kg d.s)	1286.48
NH ₄ ⁺ - N(mg/Kg d.s)	302.62
Total P, as P ₂ O ₅ (% d.s)	0.7584
K, as K ₂ O (% d.s)	3.0118
Ca as CaO (% d.s)	4.7107
Mg as MgO (% d.s)	2.1266

⁷ Council Directive 91/271/EEC concerning urban waste water treatment

Cd (mg/Kg d.s)	0.4739
Cr (mg/Kg d.s)	8.1537
Cu (mg/Kg d.s)	64.2264
Ni (mg/Kg d.s)	10.8509
Pb (mg/Kg d.s)	34.0357
Zn (mg/Kg d.s)	132.8445

The water content of compost is 36.1% which indicates that no intensive biological process can take place in such a low moisture level. In addition the ideal level of water content of the end product ranges from 35% to 45% [2, 3] and in any case it should not be lower than 25% since composts acquiring such low moisture content cannot be easily incorporated into the soil [43]. The pH was near neutral (7.31) indicating a good quality compost and within the suggested range of 6 to 8.5 as has been reported by several studies [2, 3, 5]. The total carbon was subjected to an overall 15.03% reduction throughout the 1st composting process while the remaining carbon content at the end of the process was 28.84% which is considered to be a good value [20].

With respect to the ammonium and nitrates concentration during the 1st composting trial, Figures 8 and 9 showed that when the substrate passed from the thermophilic stage to a second mesophilic, the mesophilic microorganisms that convert ammonium to nitrates flourished. The appearance of significant quantity of nitrates at the final day (886.48mg/kg) in conjunction to low ammonium concentration (302.62mg/kg) is an indicator of a maturing compost in which aerobic conditions were maintained during composting. Therefore the ratio of ammonium over nitrates to the end product can be used as indicator to assess the degree of maturity. In the case of the 1st composting trial the acquired ration was 0.24 while a ratio lower than 0.5 is indicative of mature compost [72].

During bioconversion of the materials, carbon content decreases resulting in the reduction of C:N ratio at the end of the composting process. The reduction as explained is mainly attributed to the loss in total dry mass due to losses of carbon as CO₂. Maintaining C:N ratio after composting is also important to determine the value of finished compost as soil amendment for crops. Compost with high C:N can cause

nitrogen immobilization upon amendment to soil while too low C:N ratio can cause ammonium toxicity [44, 45]. Researchers have suggested various ideal C:N ratios from lower than 12 to lower than 25 [46, 47, 48] but the optimal value is often dependent on the initial feedstock [49]. Therefore the 10.84 ratio obtained in the 1st composting trial was considered to be satisfactory taking into consideration the composition of the raw material to be composted. Nitrogen, phosphorous and potassium levels (NPK value) in the finished compost are also important in determining the quality of compost, since those elements are essential nutrients for plant growth [50]. According to Iyengar and Bhave the nitrogen, phosphorous and potassium (NPK) contents for compost should be more than 1% each [51] while a typical nutrient breakdown of finished compost is given in Table 9 according to which the nutrient concentrations in composts varies depending on the raw composted material

Table 9: Typical nutrient composition of finished compost

Nutrient	% Dry Weight
Nitrogen (N)	1% to 4.5% ^[52]
Potassium (K ₂ O)	0.5% to 1% ^[52] 1.05% to 2.96% ^[2, 4, 53]
Phosphorous (P ₂ O ₅)	0.8% to 1% ^[52]
Calcium (Ca)	2% to 3% ^[52] 2.3% to 5% ^[4]
Magnesium (Mg)	2% to 3% ^[52] 0.3% to 1.9% ^[2]

According to the aforementioned literature review and the data obtained (Table 8) the nutrients level of the end product resulting from the 1st composting trial appeared to be sufficient for plant growth.

One of the most important causes of concern in the application of compost to land is the presence of high concentrations of heavy metals, which can penetrate the soil and may affect plant health and growth, soil properties and micro-organisms, livestock and human health. Numerous heavy metals are present in sewage sludge therefore the application to the soil of composted material which originates from sludge

decomposition can pose an indirect risk to human health; due to the possibility of pollutants migration to groundwater, or their accumulation in plants. The most important heavy metals which are present in sludge are the following: lead (Pb), zinc (Zn), cadmium (Cd), chromium (Cr), copper (Cu), and nickel (Ni) [54]. The concentration of the aforementioned heavy metals in the end product resulted from the 1st composting trial has been evaluated as shown in Table 8. However there is not an EU legal piece of work that specifies limit values for heavy metals concentrations in compost that can be applied on land. Nevertheless there are several different national standards that have been set from various European countries and an EU working document on biological treatment of biowaste compost products. These suggested heavy metal limit values for compost application on land are presented in Tables 10 to 13.

Table 10: Recommended Metal Limits for Heavy Use Rates of Compost for Vegetables, with Typical Soil Levels (German standards)

Element	Max. Conc. Recommended for Intensive Compost ^a [mg/kg]	Typical Values for soils ^b [mg/kg]
Pb	75	12 - 100
Cu	50	3 - 20
Zn	200	14 - 125
Cr	75	5 - 100
Ni	30	4 - 50
Cd	0.75	0.3 - 0.7
Hg	0.5	0.05 - 0.40

a. German Hort. Assoc

b. source: BodSch (1998) Bundes-Bodenschutzverordnung. (Federal Soil Protection Rule) in Bodenschutz (Soil Protection) Erich Schmidt Verlag Berlin

Table 11: Heavy metals limit compared: EC states versus U.S.A.

Metal	EU- Range [mg/kg]	USA [mg/kg]
Cd	0.7 - 10	39
Cr	70 - 200	1,200
Cu	70 - 600	1,500
Hg	0.7 - 10	17
Ni	20 - 200	420
Pb	70 - 1,000	300
Zn	210 - 4,000	2,800

Table 12: Heavy metals limits (mg/kg) for European countries which do have compost regulations

Elements	A	A ^b Class 2 ^c	B Agr	B Park	CH	DK	F	D	I	NL	NL	SP
As	-	-	-	-	-	25	-	-	10	25	15	-
B	100	-	-	-	-	-	-	-	-	-	-	-
Cd	4	1	5	5	3	1.2	8	1.5	1.5	2	1	40
Cr	150	70	150	200	150	-	-	100	100	200	70	750
Co	-	-	10	20	25	-	-	-	-	-	-	-
Cu	400	100	100	500	150	-	-	100	300	300	90	1750
Pb	500	150	600	1000	150	120	800	150	140	200	120	1200
Hg	4	1	5	5	3	1.2	8	1.0	1.5	2	0.7	25
Ni	100	60	50	100	50	45	200	50	50	50	20	400
Se	-	-	-	-	-	-	-	-	-	-	-	-
Zn	1000	400	1000	1500	500	-	-	400	500	900	280	4000

a. Country Codes: **A** Austria; **B** Belgium; **C** Canada **DK** Denmark; **F** France; **D** Germany; **I** Italy; **NL** Netherlands; **SP** Spain; **CH** Switzerland

b. Calculated on 30% Organic Matter basis

c. NOTES: Class-2 as Versus Class 1; Agr -Agricultural use; Park= Horticultural use.

Table 13: Metal quality standards for compost and stabilised biowaste [43]

Parameter (mg/kg dm)	Compost Class 1*	Compost Class 2*	Stabilised Biowaste*
Cd	0.7	1.5	5
Cr	100	150	600
Cu	100	150	600
Hg	0.5	1	5
Ni	50	75	150
Pb	100	150	500
Zn	200	400	1500

*: normalised to an organic matter content of 30%

compost products must comply with the certain environmental quality classes with respect to their heavy metals content

The heavy metal concentrations in compost resulting from the 1st composting trial was generally low and did not exceed the suggested limits presented above with the exception of Cu⁸ values in comparison to the strict German Standards (Table 10) for maximum heavy metal concentrations for intensive compost. In addition compost

⁸ German standards Cu<50mg/kg, 1st compost = 64.2mg/kg

produced from the 1st trial is classified as first class compost based on the metal quality standards for compost and stabilised biowaste given in Table 13. Furthermore, by comparing heavy metal concentration in the initial raw material (Table 2) to that of compost (Table 8) it appears that sewage sludge composting using green waste as bulking agent may dilute heavy metal in the mass to obtain levels of trace metals sufficiently low to be used in agriculture [55]. However reports have showed that composting may concentrate or dilute the heavy metals content of sewage sludge [56, 57].

Heavy metals speciation

Most studies related to heavy metal concentration in compost examine only the total heavy metal concentration in compost. However, it is also important to evaluate the percentage of heavy metals that is dissolved in water and in weak organic acids, such as potassium nitrate (KNO₃) and Ethylene Diamine Tetraacetic Acid (EDTA), especially when compost is to be applied for agricultural purposes since in that way we can estimate the accumulation level of heavy metals in plants. Table 14 and Figures 11 and 12 present in tabular and graphical form the level of heavy metals that is dissolved in water, KNO₃ and EDTA. In water the soluble forms of metals are extracted and represent the free ions of heavy metals absorbed by plants [58]. KNO₃ is used to determine the exchangeable forms of heavy metals. The absorption of the exchangeable forms of heavy metals takes place through metal ion exchange with another ion. Finally, EDTA is used to determine the complex heavy metal forms [58].

Table 14: Heavy metals speciation in compost resulted from the 1st composting trial [mg/kg]

Heavy Metals	Total	Dissolved in H₂O	Dissolved in KNO₃	Dissolved in E.D.T.A	Total dissolved metals	Total dissolved [%]
Cd	0.4739	0.0037	0.0104	0.0154	0.0295	6.21
Cr	8.1537	0.0083	0.1366	0.0292	0.1741	2.13
Cu	64.2264	0.5915	0.8349	1.5954	3.0218	4.70
Ni	10.8509	0.1048	0.2375	0.3207	0.6630	6.11
Pb	34.0357	0.0917	0.1637	0.8711	1.1265	3.31
Zn	132.8445	1.1759	1.5849	3.672	6.4328	4.84

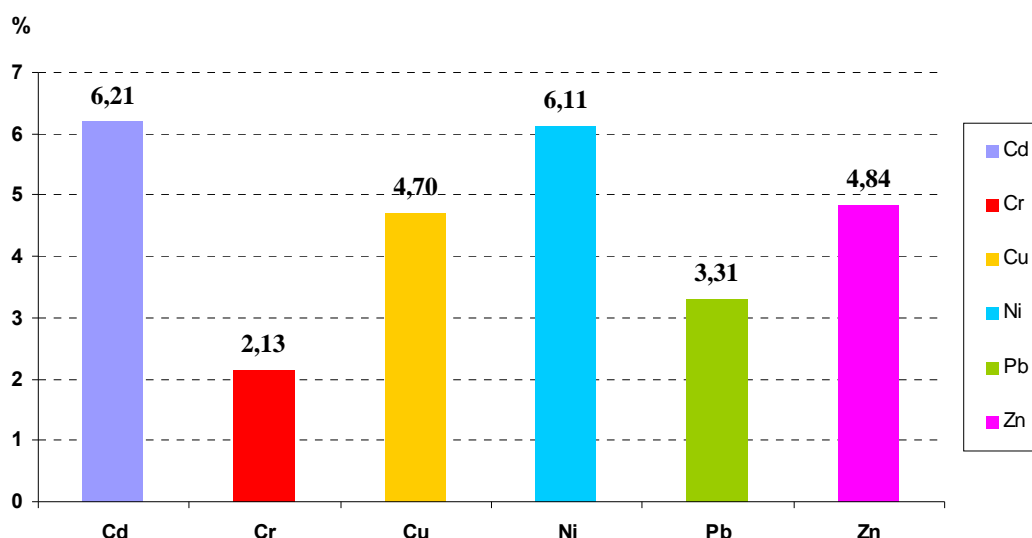


Figure 11: Percentages of total dissolved heavy metals in compost resulted from the 1st composting trial

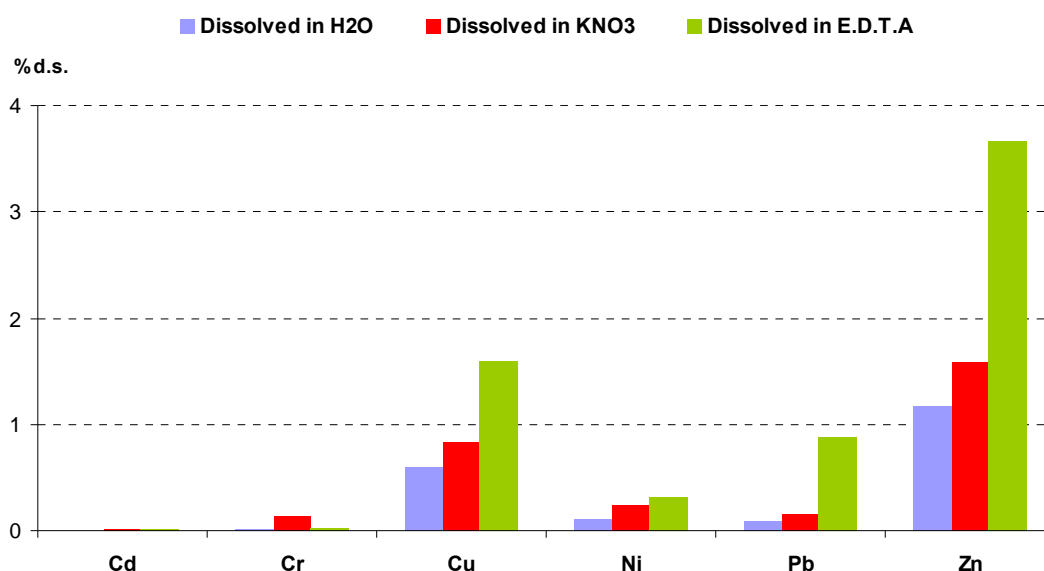


Figure 12: Heavy metals content [% d.s.] that is dissolved in water and in weak organic acids in compost resulted from the 1st composting trial

According to Table 14 and Figure 11, Cd and Ni had the highest percentages of total heavy metal dissolution, rated at 6.21% and 6.11% respectively followed by Zn and Cu with 4.84% and 4.70% respectively but in real values the concentrations is low rating from 0.0295mg/kg to 6.4328mg/kg . With respect to heavy metal content that is dissolved in water, KNO₃ and EDTA, Zn, Cu, Ni, and Pb were the heavy metals with the highest quantity of potential accumulation in plants as presented in Figure 12. This is in accordance to the cumulative research in Europe for the agronomic use of compost, according to which heavy metals tend to accumulate in soil and plants in the following order: Zn > Cu > Pb = Cd > Ni > Cr [59, 60].

Biological assays

To estimate the reduction of the pathogenic microorganisms analyses have been performed to evaluate their density prior and after the 1st composting trial. The density of total coliforms, faecal coliforms and helminth eggs has been measured initially as shown in Table 4 while pathogens density of the finished compost is shown in Table 15.

Table 15: Micro-biological parameters in compost produced in the 1st composting trial

Micro-biological parameters	Compost
Total coliforms Log10 MPN/10g DS	0.60
Faecal coliforms Log10 MPN/10g DS	< 0.1
Helminth eggs /10g DS	< 1

The elimination or inactivation of pathogenic microorganisms in composts depends upon the time/temperature conditions maintained during composting. According to Stentiford, temperatures higher than 55°C favour sanitation, values between 45°C and 55°C favour biodegradation, and those between 35°C and 40°C favour microbial diversity [61]. The thermophilic temperatures are preferred in the composting materials, because they destroy the majority of pathogenic microorganisms. A temperature of 55°C should kill human pathogens and most plant pathogens as well. It appears that in the 1st composting trial the total and faecal coliforms as well as helminth eggs have diminished at the end of the composting process as shown in Table 15 in comparison to Table 4. Therefore it can be assumed that the temperature/time regime during the thermophilic composting phase was responsible for the reduction of the pathogenic microorganism density to the end product [62]. The elevated temperatures (>55°C) obtained for approximately 7 consecutive days, as shown in Figure 2 (6th to 12th day), had an influence on the presence of pathogenic microorganisms and it seems that the temperature reached during the composting process was responsible for the elimination of the pathogenic microorganisms [12, 13]

There is no legal document which regulates the level of pathogenic microorganisms in compost which is considered safe for compost application to land. However, in order to reduce potential health risks related to pathogens, several national regulations have added limitations concerning the pathogen content for sludge applied to land. All

countries with statutory standards in place, with the exception of the Netherlands, where only a limited version exists, have testing criteria in place for the content of pathogens (Table 16). Pathogen testing usually involves testing for the presence of specific micro-organisms. Voluntary systems also often have such tests in place (Sweden, Netherlands, United Kingdom, Canada, Australia and, indirectly, New Zealand). These tests support the process-oriented ‘temperature–time’ regimes in seeking to ensure a hygienic product.

Table 16: Requirements concerning pathogens in EU countries [14]

Austria	Statutory, dependent on area of application
Belgium Flanders	Statutory, indirect process control
Belgium Walloon	Statutory, indirect process control
Belgium Brussels	Statutory, indirect process control
Denmark	Statutory
Finland	Only remark may not contain to a harmful extent
France	Statutory no harmful micro-organisms which may endanger man, animals or the environment
Germany	Statutory process and product tests
Greece	Statutory no Enterobacteria should be detectable
Ireland (licensing)	(under licensing regime) for human and plant pathogens
Italy	Statutory
Luxembourg (licensing)	Statutory process test and product test
Netherlands	Voluntary product tests
Portugal	No
Spain	Statutory product test
Sweden	Voluntary – product test
UK (Composting Association)	Product test – for 2 human pathogen indicator species
Canada	CCME (Statutory) and BNQ (Voluntary) set limits for faecal coliforms and absence of Salmonellae
USA	Statutory – product test
Australia	Through state or federal guidelines on biosolids
New Zealand	Voluntary – not explicitly set only through cross-reference to DoH regulations

5.1.10. Preparing the system for the implementation of the next composting trial

After the removal and collection of the compost the electromechanical components of the system were monitored and maintained while the whole system was cleaned up thoroughly. The maintenance and monitoring of the system were carefully planned and carried out according to the instructions specified in the operation and maintenance manual.

5.2. Composting using primary sludge green waste and manure - 2nd trial

In this section the experimental results obtained from the 2nd composting trial are shown. The feedstock includes primary sludge from El Jadida city, green waste and manure. Initially the feedstock was chosen and the composition of the raw material was determined. Next the operational conditions of the bioreactor were set taking into consideration the experience and results obtained in the 1st composting trial in order to improve and optimize the overall process, such as hydration, aeration and temperature control. Composting samples were taken at regular time intervals from the substrate and leachates for analyses and evaluation of the process. Samples were also collected from the end product for detailed analyses such as physicochemical analyses and biological assays.

5.2.1. Sampling and analysis of the raw material

The rate at which materials compost as well as the quality of the final composted organic material, are largely depended on the initial selection and mixing of raw materials. Table 17 presents the physicochemical characteristics of the primary sludge, sheep manure, sugar beet leaves and straw residues that were used as feedstock for the operation of the 2nd composting trial. As has been stated, the characterisation of the raw material that is used as feedstock into the in-vessel composting bioreactor is essential for the evaluation of the aerobic composting process and thus to the evaluation of the end products since the raw material determines a priori the physical, chemical and biological conditions involved in composting processes.

Table 17: Physicochemical parameters of the raw material used in the 2nd composting trial

Parameter	Primary sludge	Sheep Manure	Sugar beet leaves	Straw
Dry Solids (% d.s)	32.16	84.37	53.74	90.27
pH	6.3	8.5	7.2	7.1
Total C (% d.s)	11.03	47.71	58.60	59.08
Total N (% d.s)	1.91	2.83	3.43	0.94
C:N ratio	5.77	16.86	17.08	62.85
Total P, as P ₂ O ₅ (% d.s)	1.3942	0.9310	0.2470	0.1721

K, as K ₂ O (% d.s)	0.9670	4.2683	3.4673	1.9890
Ca as CaO (% of d.s)	4.5235	6.8071	3.4418	1.1122
Mg as MgO(% of d.s)	2.2073	1.7388	1.5807	0.5666
Cd (mg/Kg d.s)	0.8218	0.5937	0.1590	0.1305
Cr (mg/Kg d.s)	25.2105	10.4570	0.5719	0.6012
Cu (mg/Kg d.s)	135.2713	17.3205	12.2520	6.6361
Ni (mg/Kg d.s)	26.9106	11.1481	0.9365	1.0037
Pb (mg/Kg d.s)	63.3828	2.8133	0.3043	0.3260
Zn (mg/Kg d.s)	251.8231	34.8637	31.9314	23.909

According to Table 17 the primary sludge used in the 2nd composting trial acquired high moisture content of 67.84% since the collected sludge did not undergo dewatering. On the other hand sheep manure, sugar beet leaves and straw residues had much lower water content of 15.63%, 46.26% and 9.73% respectively which could balance the water level of the substrate when mixed with primary sludge. The pH of the raw material ranged from 6.3 to 8.5 which is considered satisfactory for the development of composting processes since compost microorganisms operate best at pH values in the range of 6 to 8.5 [2, 3, 4, 5, 6]. With respect to the total carbon content straw residues and sugar beet leaves acquired the highest carbon content, 59.08% and 58.60% respectively, followed by sheep manure (47.71%). Sludge acquired a carbon content of 11.03%. In comparison to the other raw materials the nitrogen content in straw residues, proportionally to the total carbon content, was lower leading to a C:N ratio of 62.85. Thus, appropriate blending of these materials will have C:N and moisture values suitable for the development of microorganisms involved in composting. Apart from the chemical properties sugar beet leave and straw residues make also good quality structuring agents for mixing with sludge by increasing the porosity of the substrate and favouring aeration within the bioreactor. In addition, the NPK value of the raw material was considered sufficient for the purposes of composting in protein synthesis of the microorganisms. With respect to the heavy metal content the primary sludge used in the 2nd composting trial did not have limiting heavy metal values for the composting process. Zn and Cu were the trace-elements acquiring the highest concentration however they did not exceed the limits for sludge application in agricultural land as has been set by Directive 86/278/EEC (Table 3).

Table 18 shows that the primary sludge used in the 2nd composting trial contained high number of pathogenic microorganisms which constitutes a health hazard and if they are to be used for agricultural purposes without being submitted to a proper treatment process, such as composting, there is a strong risk of contamination [12, 3, 14,15].

Table 18: Micro-biological parameters of primary sludge used in the 2nd composting trial

Micro -biological parameters	Primary sludge
Total coliforms Log10 MPN/10g DS	7.84
Faecal coliforms Log10 MPN/10g DS	7.69
Helminth eggs /10g DS	26.27

5.2.2. Composition of the composting raw material

The feedstock material of the 2nd composting trial comprised of primary sludge from El Jadida city, sheep manure, straw residues and sugar beet leaves. The quantities of each material used are given in Table 19.

Table 19: Composition of the mix used for composting in the 2nd composting trial

Raw material	Kg w.w.
Primary sludge	520
Sheep manure	250
Straw	50
Sugar beet leaves	490

5.2.3. Preparation of the composting raw material

The sugar beet leaves were shredded to size 2 to 4 cm using a commercial shredder type MTD 118E prior its supply into the bioreactor. After the green material had been shredded it was then weighted and packed ready to be used. The primary sludge, sheep manure and straw residues were also weighted and packed ready to be used. The weighted and packed raw materials were then mixed manually using shovels and ready to be fed into the bioreactor.

5.2.4. Feeding the in-vessel bioreactor

The feeding of the bioreactor was performed using the mixture prepared as indicated in paragraph 5.2.3 via the conveyor belt in which the feedstock was loaded manually using shovels.

5.2.5. Composting process control

Composting process control involves all the actions that had been planned and carried out with respect to the operational conditions of the in-vessel bioreactor.

5.2.5.1. Programming the plc of the in-vessel bioreactor

The operation of the agitation, hydration and aeration systems was performed by the PLC automatic control system. These operations can also be performed manually. The programming of each operation during the 2nd composting trial is given below.

Programming the frequency and duration of the substrate's agitation:

The motor was programmed to operate 4 times per day (every 6 hours) for 5 minutes each time for the agitation of the substrate.

Programming the frequency and duration of the substrate's aeration:

The fan was programmed to operate 4 times per day (every 6 hours) for 5 minutes each time.

Programming the frequency and duration of the substrate's hydration:

The hydration system operated manually during the 2nd composting trial although it could be programmed to operate automatically. The substrate was subjected to hydration whenever it was considered appropriate according to its moisture content that had been recorded from the daily measurements. The duration of the hydration operation as well as the water flow were also adjusted according to the moisture content of the substrate that had been recorded from the daily measurements

5.2.5.2. Measuring the temperature of the substrate

Temperature is considered to be one of the most important factors during composting process since composting is determined by the temperature profile. Changes in temperature are commonly used as a measurement of microbiological activity underlying the composting process and thus determining the stability of the organic material. Figure 14 shows the temperature evolution during the 2nd composting trial.

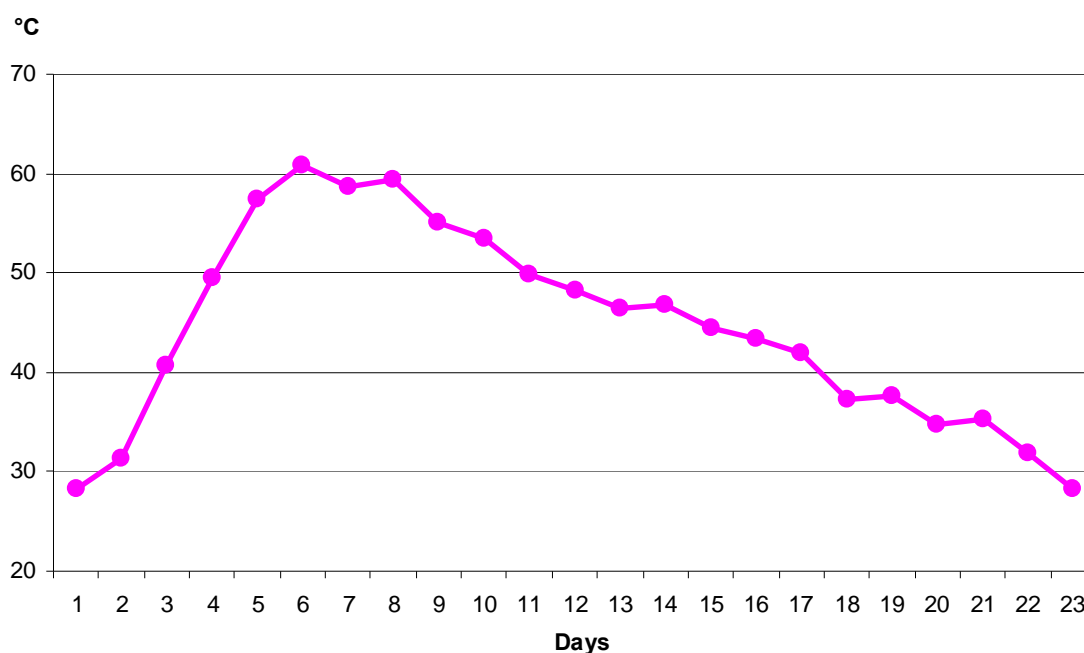


Figure 13 Temperature profile during the 2nd composting trial

According to Figure 13 the substrate passed from an initial mesophylic phase ($<40^{\circ}\text{C}$) to a thermophylic stage after the 3rd day of the initiation of the composting process. Due to the early decomposition of the soluble, readily degradable compounds which are present mainly at the primary sludge compounds, large amounts of heat were released and caused the temperature to rise rapidly and reached to a maximum of 60.9°C on the 6th day. Elevated temperatures ($>50^{\circ}\text{C}$) were maintained in the bioreactor for eight continuous days (4th to 11th day) which is sufficient time for the sterilisation of the substrate from potential pathogenic microorganisms that exist in the composting material. Only after all substrate, capable of promoting microbial activity, had been used up did the temperature begin to fall to reach a second mesophylic phase on the 17th day of the process while the maturation process took place during this last stage of composting.

5.2.5.3. Measuring the moisture of the substrate

Low moisture values (<40%) indicate early dehydration of the substrate which arrests the biological process giving a physically stable but biologically unstable compost while high moisture quantities (>60%) interfere with the aeration by clogging the pores which favour undesirable anaerobic activities. Figure 15 shows the moisture content evolution during the 2nd composting trial.

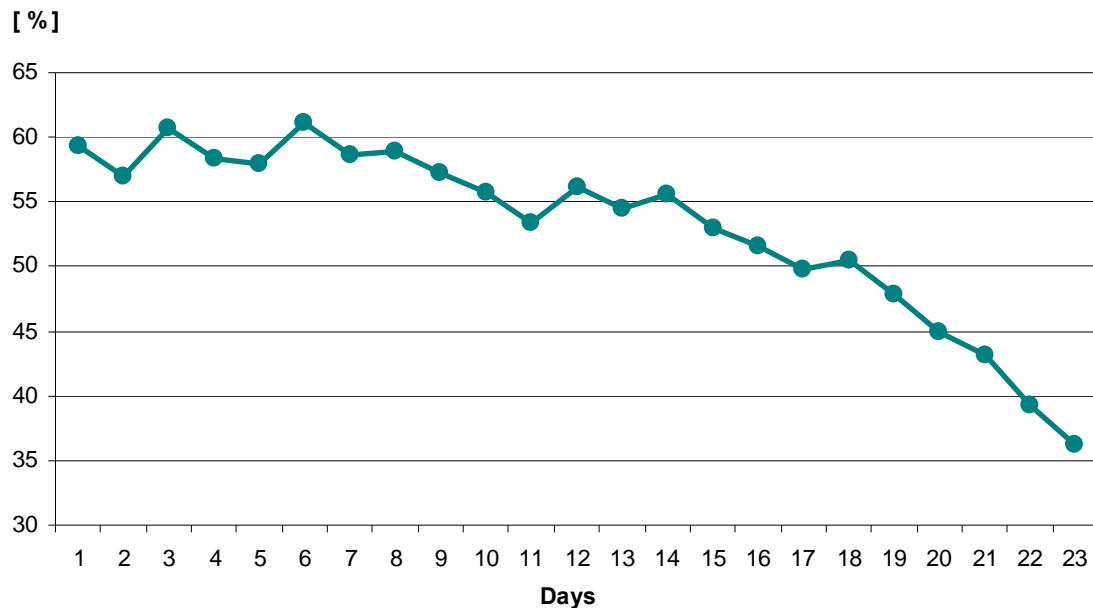


Figure 14: Moisture content profile during the 2nd composting trial

According to Figure 14, the initial mixture of the 2nd composting trial had a moisture level of 59.3%. The moisture content was sustained at optimal levels around 50 to 60% for the first 18th days of the process due to the hydration of the substrate and its sufficient agitation and aeration. At the final stage of composting the water quantity was gradually decreased reaching at 36.2% at the final day of the process on the 23rd day.

5.2.5.4. Measuring the oxygen content of the substrate

The readily degradable components of the raw materials are metabolised during the initial period of composting. Therefore the production of heat and the need for oxygen are greatest at the early stages and then steadily decrease. Without a constant supply of oxygen, the process will slow down and if there is insufficient oxygen, the process can become anaerobic. As has been mentioned composting process takes place even

in low oxygen content (5%) however, oxygen concentrations greater than 10% are consider optimal for maintaining aerobic conditions and thus sustaining aerobic microbial activity. Figure 15 presents the moisture evolution during the 2nd composting trial.

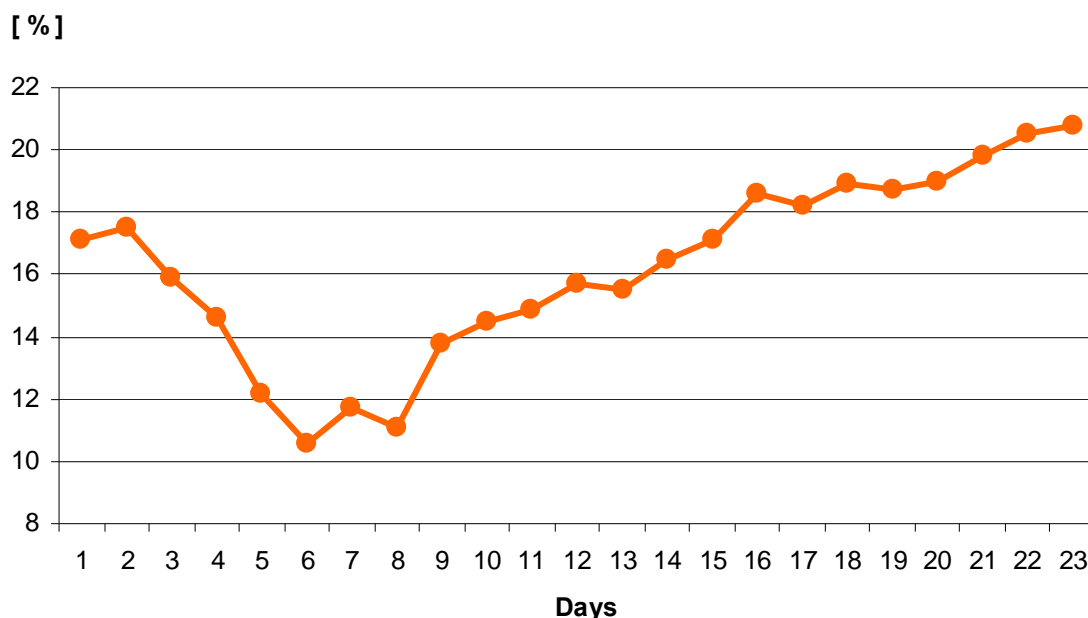


Figure 15: Oxygen profile during the 2nd composting trial

According to Figure 15 the oxygen concentration of the substrate was maintained higher than 10% throughout the duration of the 2nd composting trial. The lowest oxygen concentration values were recorded during the thermophilic composting phase due to the high microbial activity of aerobic microorganisms which consume oxygen for the decomposition of the organic matter. The increased oxygen level and the incremental tendency of oxygen concentration during composting indicate that the agitation and thus the aeration of the substrate were sufficient in promoting aerobic conditions in the bioreactor. At the latest stage of the composting process the level of oxygen exceeded the 20% indicative of the low rate of organic matter decomposition since the concentration of oxygen nearly reached that of the atmosphere (21%).

5.2.5.5. Reprogramming the plc of the in-vessel bioreactor

The plc of the in-vessel bioreactor was not reprogrammed during the 2nd composting trial.

5.2.5.6. Monitoring and maintenance during composting

Throughout the duration of the composting process the electromechanical equipment and the rest of the bioreactor's components and infrastructure were being monitored daily in order to maintain their operation status. The regular maintenance of all the electromechanical equipment of the system was performed according to the suggestions of the manufacturer (e.g. lubrication, cleaning the filter of the ventilation system, cleaning the head of the piping system, leachate removal). In addition, daily cleaning of the floor and surface of the bioreactor was performed in order to obtain high hygiene level for the personnel.

5.2.5.7. Disruption and start up of the bioreactor operation

No disruptions of the bioreactor's operation occurred during the 2nd composting trial which lasted for 23 days.

5.2.5.8. Restoration of malfunctions and maintenance of the bioreactor

No malfunctions occurred during the 2nd composting trial.

5.2.6. Sampling and analysis of the substrate

Substrate samples were taken every 4 to 7 days of the composting process and the examined parameters included the pH, total carbon, total nitrogen, nitrates ($\text{NO}_3^- - \text{N}$) and ammonium nitrogen ($\text{NH}_4^+ - \text{N}$).

5.2.6.1. pH

As mentioned the composting process can work over a wide spectrum of pH values, but a range of between 6 and 8.5 is preferred. Figure 16 presents the evolution of pH values during the 2nd composting trial.

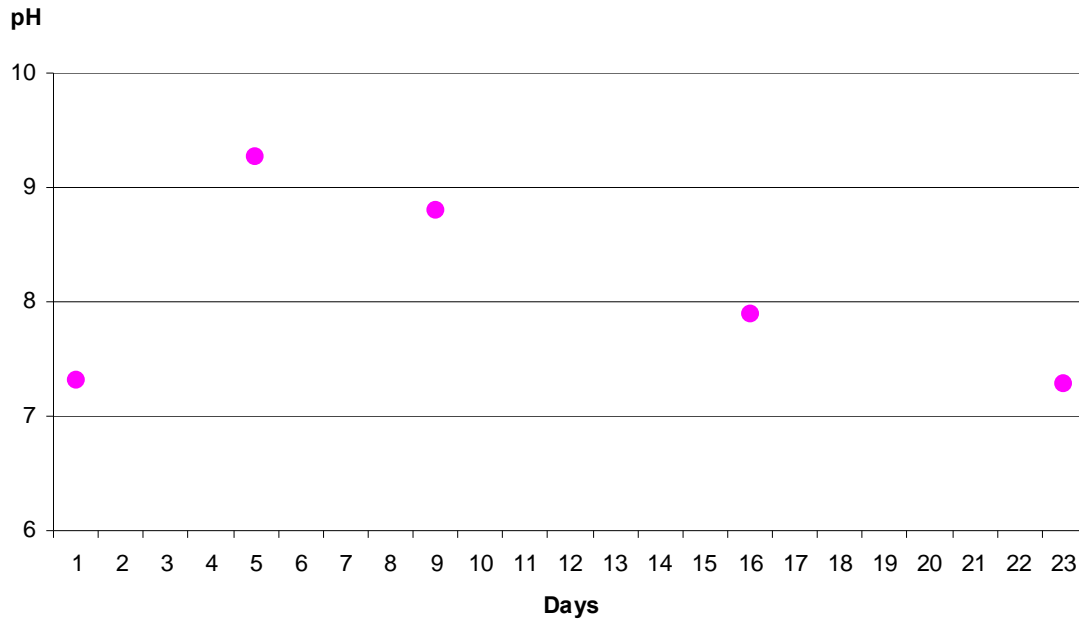


Figure 16: Changes in pH values during the 2nd composting trial

As shown in Figure 16 the pH evolution in the 2nd composting trial follows the same pattern as that of the 1st composting trial. The pH values increased during the initial stage of composting (9.27 on the 5th day) probably due to the activity of proteolytic bacteria which release ammonia when organic matter is decomposed. Then through ammonia volatilization and oxidation to nitrates the ammonia content decreased and thus the pH value dropped from 9.27 to 7.28 at the final day of the process.

5.2.6.2. Total Carbon

The mineralization process which takes place in the composting bioreactor results in lowering the total carbon content of the substrate. Figure 17 presents the total carbon content evolution during the 2nd composting trial.

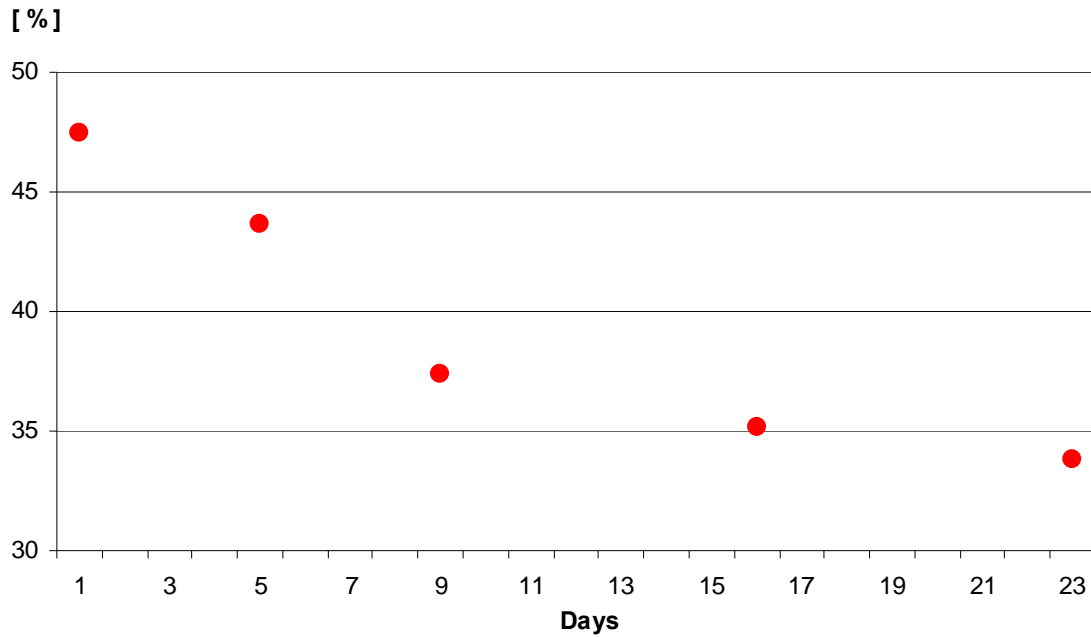


Figure 17: Changes in total carbon content [%] during the 2nd composting trial

The carbon mineralization in the 2nd composting trial was principally due to the primary sludge, sheep manure and sugar beet leaves (sucrose content) since straw residues have a more consistent structure such as cellulose and lignin type compounds which are less susceptible to microbial attack. The microorganisms therefore used the labile carbon compounds as energy source rather than the complex compounds incorporated in the bulking agent [12]. At the initial stage of composting the “available” carbon was consumed rapidly reducing the carbon content from 47.46% to 37.42% during the first nine days of the process. As composting was developing the rate in which carbon was consumed decreased due to the depletion of carbon compounds capable of promoting intensive microbial activity. At the end of the process the carbon had been reduced to 33.84% achieving a 13.62% decrease compared to the initial carbon content of the substrate. The final carbon content of the end product resulting from the 2nd composting trial is of good quality with respect to its carbon content [19].

5.2.6.3. Total Nitrogen

Figure 18 presents the total nitrogen content evolution during the 2nd composting trial.

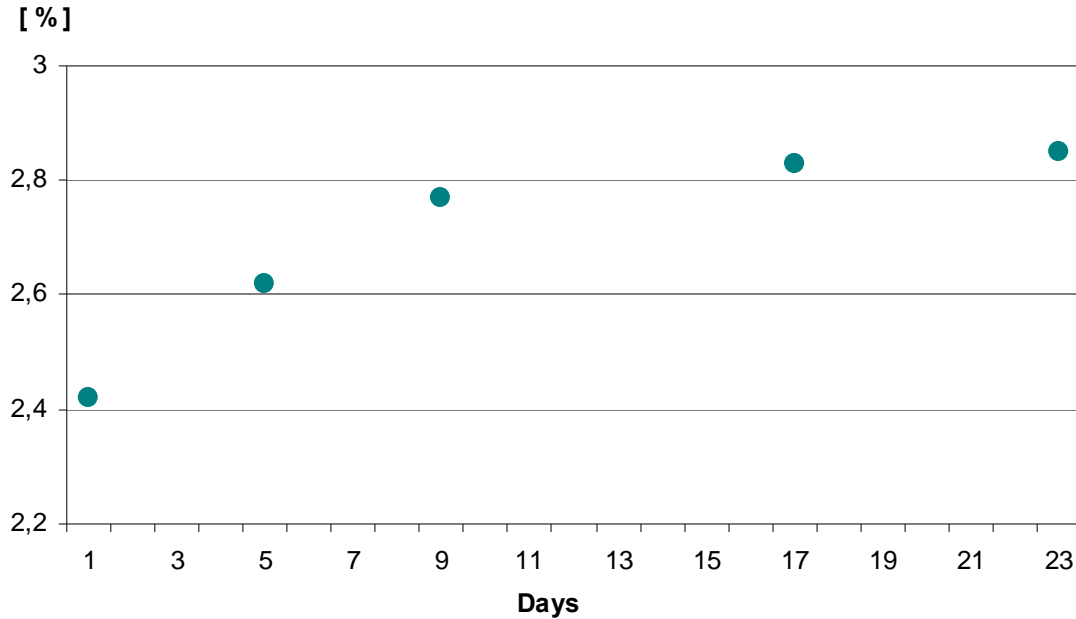


Figure 18: Changes in total nitrogen content [%] during the 2nd composting trial

As has been mentioned nitrogen decreases during the process of composting mainly through ammonia volatilization but the increase in total nitrogen content is attributed to a concentration effect as a consequence of strong degradation of organic carbon compounds which reduces the weight of the composted dry mass [3]. Therefore in dry bases the total nitrogen content presented a linear increase during the first 9 days of the process which is explained by the steep decrease of the total carbon as shown in Figure 18. Iranzo et al. reported that increase of total nitrogen content has been observed in composting processes in which the organic matter has been reduced significantly [62]. From the 9th day onwards the rate in which nitrogen content increased is reduced mainly due to the reduction rate in which carbon is consumed. Studies confirm that additional increase of nitrogen content, at the latest stage of the composting, may be acquired due to processes of non-symbiotic nitrogen fixation which occurs due to the activity of azotobacters which recover a proportion of nitrogen which has been lost as ammonia [2, 28]. The nitrogen content at the end of the 2nd composting trial on the 23rd day was 2.85% which is higher than the initial nitrogen content (2.42%).

5.2.6.4. Ammonium Nitrogen ($\text{NH}_4^+ - \text{N}$)

The evolution of the ammonium concentration of the substrate during the 3rd composting trial follows the same pattern as the 1st composting trial. Figure 19 presents the changes of the ammonium content during the 2nd composting trial.

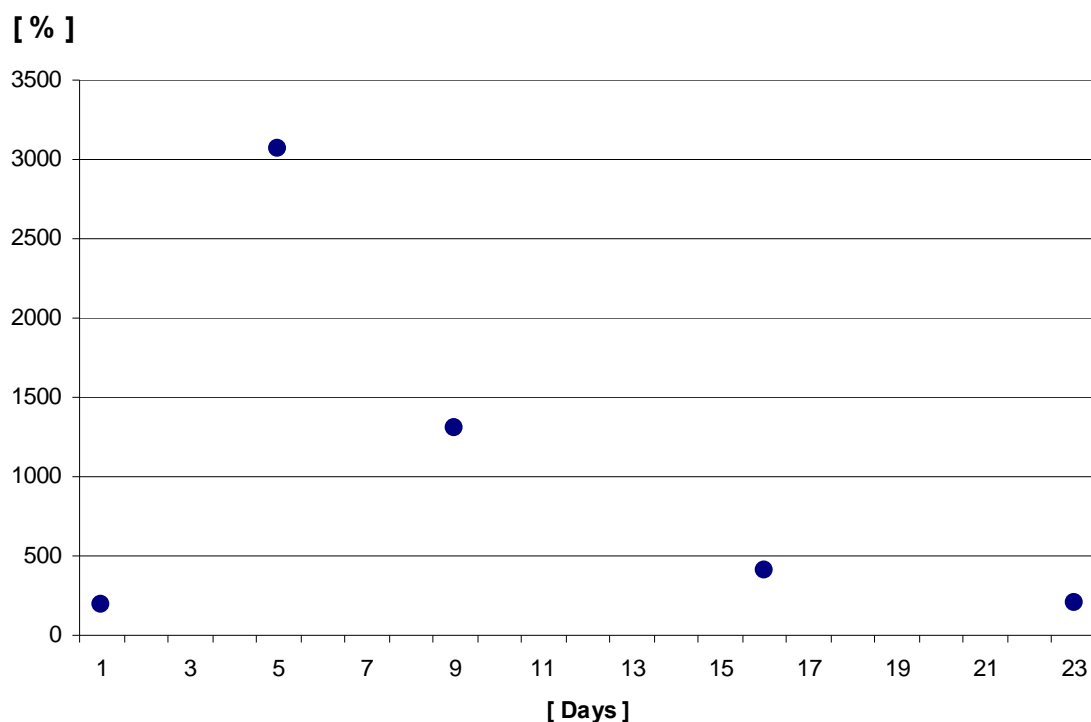


Figure 19: Changes in ammonium concentration [mg/kg] during the 2nd composting trial

Due to the activity of proteolytic bacteria ammonium was produced reaching a maximum of 3063.60mg/kg on the 5th day of the process. From that point onwards the ammonium concentration was reduced significantly due to ammonia volatilization and the stimulation of nitrification and reached at 207.73mg/kg at the final day of composting.

5.2.6.5. Nitrates ($\text{NO}_3^- - \text{N}$)

With respect to the nitrates concentration Figure 20 presents the changes of nitrates during the 2nd composting trial.

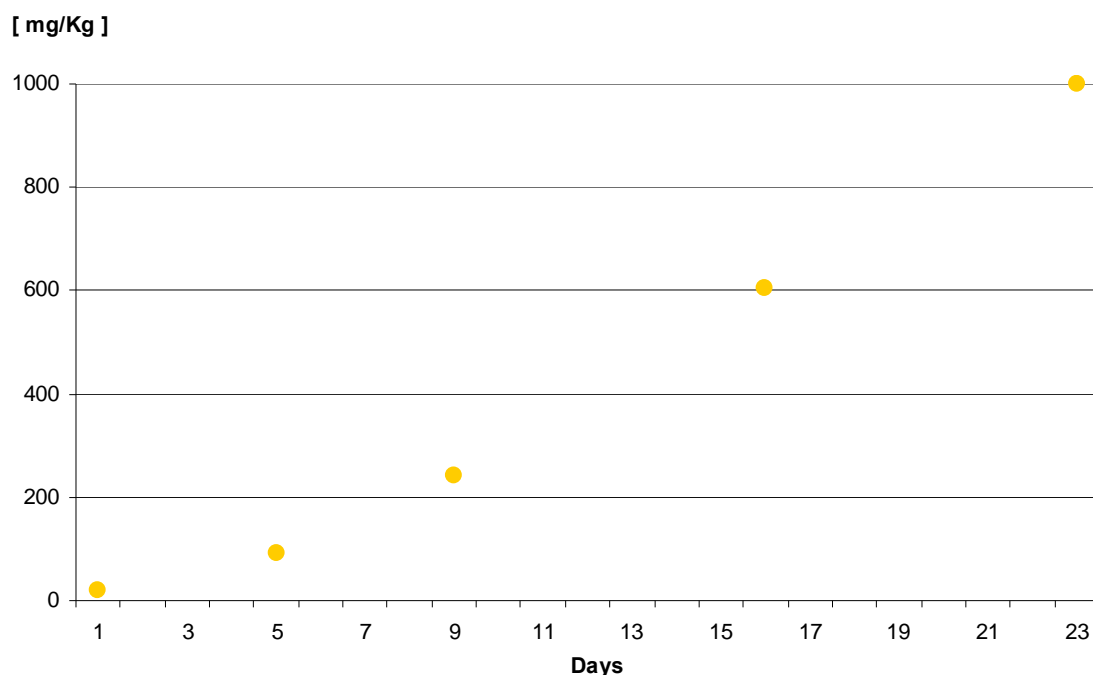


Figure 20: Changes in nitrates concentration during the 2nd composting trial

According to Figure 20 during the early stages of the process the nitrate content increased slowly (21.16mg/kg to 91.62mg/kg on the 1st and 5th day respectively) due to the heterotrophic nitrification in which ammonium is converted to nitrates. From the 5th day onwards a significant increase of nitrates concentration was observed which can be explained by the activity of autotrophic nitrobacteria which oxidize ammonia compounds into nitrates in the presence of oxygen-rich environment as shown in Figure 15. Nitrates reached at 998.43mg/kg on the 23rd and final day of the process. It must be mentioned that the rapid decrease in ammonium shown in Figure 19 did not coincide with a rapid increase in nitrates concentration. The concentration of nitrates was very low initially suggesting that nitrogen was lost during composting. Losses of nitrogen in the composting process were governed mainly by volatilization of ammonia due to the high temperature and pH values (Figures 13 and 16) of the substrate. Gaseous ammonia and aqueous ammonium ion are in equilibrium at a pH of about 9, with higher pH's forcing more ammonium into the gas form. Agitation and aeration rate may have also affected the rate of ammonia volatilization [30, 63].

5.2.6.6. C:N Ratio

The course of decomposition and stabilization of the organic matter during composting is affected by the presence of carbon and nitrogen. A ratio of about 30

parts carbon for each part of nitrogen is considered as an ideal ratio for composting process. Figure 21 presents the carbon to nitrogen ratio evolution during the 2nd composting trial.

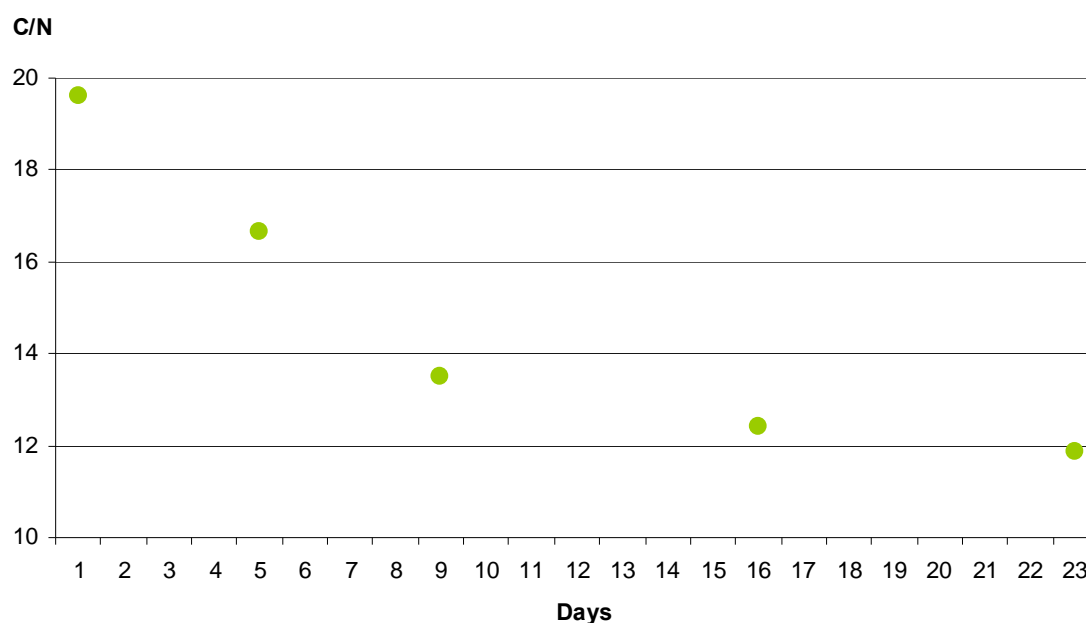


Figure 21: Changes in C:N ratio during the 2nd composting trial

Although the initial C:N ratio of 19.61 obtained in the 2nd composting trial is not within the optimum range, rapid composting took place as has been shown by the elevated temperatures at the early stages of the process in. According to Figure 21 the C:N ratio presented a steep decrease during the first 9 days of the process due to the rapid carbon consumption as presented in Figure 17. As soon as the readily available carbon compounds were depleted the C:N reduction rate decreased. At the end of the process the C:N ratio has decreased to 11.87 which is qualified as a good quality compost which can be applied in agricultural land. As has been mentioned earlier composts acquiring a C:N ratio higher than 20 interfere with plant nutrition since soil microflora compete with the plants roots for nitrogen [2, 15]. As has been shown the present composting trial acquires a C:N ratio lower than 20 and thus the above mentioned effect does not apply. In addition Bernal et al. suggested that one of the maturity indices in compost is a C:N ratio below 12 using sewage sludge, poultry manure and maize straw as feedstock material [64] while other studies has shown that a too low C:N ratios can cause ammonium toxicity [44,45].

5.2.7. Sampling and analysis of the substrate's leachate

Table 20 presents the evolution of the leachates parameters examined during the 2nd composting trial. The parameters examined are pH, BOD₅, ammonium, nitrates and heavy metals.

Table 20: Characteristics of leachates resulting from the 2nd composting trial

Parameter/Time	3rd day	6th day	11th day	18th day
pH	8.7	8.9	8.1	7.6
BOD (mg/L)	60582	40393	25956	13802
NH ⁴⁺ - N (mg/L)	1303.87	1885.41	709.26	613.79
NO ³⁻ - N (mg/L)	31.73	67.36	251.12	442.51
Cd (mg/L)	0.00165	0.00094	0.00061	0.00075
Cr (mg/L)	0.01825	0.01003	0.00838	0.00983
Cu (mg/L)	0.11782	0.09895	0.08190	0.10737
Ni (mg/L)	0.03356	0.02638	0.02327	0.02770
Pb (mg/L)	0.04565	0.04295	0.03888	0.03250
Zn (mg/L)	0.27343	0.26764	0.29014	0.32614

The pH values of leachates change in the same manner as the substrate's pH values since the physicochemical conditions involved in composting were the same in the liquid and solid state. The pH in leachates increased during the initial stage reaching to a maximum of 8.9 and then it was gradually reduced acquiring a near neutral pH of 7.6 at the end of the process. The initial sample of leachates derived from the 2nd composting trial acquired an increased initial BOD₅ value of 60.6g/L on the 3rd day. As the process kept developing the need for oxygen in leachate decreased and thus the BOD₅ values declined and reached at the 18th day the value of 13.8g/L which is approximately 1/4 of the initial BOD₅ value. Leachates acquired at the initial stage of composting a light dark colour which was gradually becoming darker as composting was developing. This is indicative of the presents of humic/fulvic and other complex compounds which are dark coloured, hydrophilic organic compounds. Ammonium and nitrates in leachates followed the same pattern as the ammonium and nitrates of the substrate indicating that the composting process was well monitored since the conditions were in favor of nitrification. Ammonium concentration in leachates showed an increase during the first days of composting reaching to a maximum of 1885.41mg/L on the 6th day. From that day onwards ammonium concentration was

reduced and on the 18th day ammonium concentration was 613.79mg/L. Nitrates in leachates presented a significant increase throughout the duration of the 2nd trial. On the 3rd day their concentration was limited at 31.73mg/L while on the 18th day it has been increased approximately 14 times reaching at 442.51mg/L. Table 20 shows also that heavy metals such as Cd, Cr, Cu, Ni, Pb and Zn were present in leachates throughout the process. The metal concentrations were much below the indicative values for wastewater disposal as shown in Table 7.

According to BOD₅, ammonium and nitrates values obtained for the leachates of the 2nd composting trial it can be concluded that leachates had a heavily polluted organic and inorganic load which needs to be treated before its final disposal. The quantity of leachates production during the 2nd trial was limited and stored to an impermeable reservoir for sufficient period of time to be stabilized at lower values through aeration. Although composting can be assessed for sludge treatment and reuse through land application, leachates remain an environmental problem since the treatment of solid waste through composting results in transferring the problem from solid to liquid phase. Leachates must be treated accordingly in order to be disposed to a water recipient based on the suggested standards shown in Table 7. However Directive 91/271/EEC seeks to harmonise measures relating to the treatment of wastewater at Community level in order to protect the environment from any adverse effects due to discharge of such waters.

5.2.8. Removal and collection of the composting end product

The removal and collection of compost was performed on the 23rd day of the composting process through the removal portals that are situated at the bottom part of the bioreactor. To facilitate the discharge of the end product the agitation system was operated for small intervals. The compost was collected manually using shovels and it was spreaded in an open area outside the building where the bioreactor is installed where it remained for five days to further reduce its moisture content (during that time the end product was regularly agitated manually). Finally the compost was stored in order to be used to the open field experiments.

5.2.9. Sampling and analysis of the produced compost

In order to evaluate the quality of the end product resulting from the 2nd composting trial a series of parameters were examined. Table 21 presents the physicochemical properties of the compost produced from the raw material shown in Table 19 which acquire the characteristics presented in Table 17.

Table 21: Physicochemical parameters of the compost produced in the 2nd composting trial

Parameter	Compost
Dry Solids (% d.s)	63.8
pH	7.28
Total Carbon (% d.s)	33.84
Total Nitrogen (% d.s)	2.85
C:N Ratio	11.87
NO ₃ - N (mg/Kg d.s)	998.43
NH ₄ ⁺ - N(mg/Kg d.s)	207.73
Total P, as P ₂ O ₅ (% d.s)	0.9264
K, as K ₂ O (% d.s)	3.9505
Ca as CaO (% d.s)	5.4785
Mg as MgO (% d.s)	2.0931
Cd (mg/Kg d.s)	0.59735
Cr (mg/Kg d.s)	11.19308
Cu (mg/Kg d.s)	54.17476
Ni (mg/Kg d.s)	11.96504
Pb (mg/Kg d.s)	20.03207
Zn (mg/Kg d.s)	109.24231

The water content of the second compost is 36.2% which is within the desirable range (35% to 45%) and not lower than 25% since composts acquiring such low moisture content cannot be easily incorporated into the soil [2, 3, 43]. The pH was near neutral (7.28) indicating a good quality compost and within the suggested range of 6 to 8.5 [2-6]. The total carbon was subjected to an overall 13.62% reduction throughout the 2nd composting process while the remaining carbon content at the end of the process was 33.84% which is considered to be a good value [20].

With respect to the ammonium and nitrates concentration during the 2nd composting trial, Figures 19 and 20 showed that nitrates increased significantly reaching to a maximum of 998.43mg/kg at the final day of the process while at the same time ammonium concentration presented a steep decrease and amounted at 207.73mg/kg at the end of the process. In the case of the 2nd composting trial the acquired ammonium to nitrates ratio was 0.21 which is lower than the suggested 0.5 ratio indicative of mature composts [72].

As has been shown in section 5.2.6.6 the optimal C:N value of finished compost varies indicating values that range from lower than 12 to lower than 20 while too low C:N ratios can be toxic. It must also be mentioned that the ideal C:N ratio is usually dependent upon the initial feedstock [49]. Therefore the final C:N ratio of 11.87 obtained at the final day of the 2nd trial is considered to be satisfactory.

With respect to the nutrient content, the compost produced in the 2nd trial was rich in nourishing elements/macroelements which are needed in great quantities in order to promote plant growth and seed germination. The end product acquired an NPK value higher than 1% for each macroelement and it was in accordance with the typical nutrient breakdown of finished compost given in Table 9.

As has been mentioned the most important heavy metals which are present in sludge are Cadmium (Cd), Chromium (Cr), Copper (Cu), Nickelium (Ni), Lead (Pb) and Zinc (Zn) (WHO, 2005). According to Table 21 the heavy metal concentration of the produced compost is within the range of EU states heavy metal suggested limits (Table 11) and complies even to the strict German standards (Table 10) for maximum heavy metal concentrations for intensive compost with the exception of Cu⁹. In addition the compost produced from the 2nd trial is classified as first class compost based on the metal quality standards for compost and stabilised biowaste given in Table 13.

⁹ German standards Cu<50mg/kg, 2nd trial compost = 54mg/kg

Heavy metals speciation

Furthermore analysis of heavy metal speciation in compost was implemented (Table 22, Figures 22 and 23) in order to estimate the accumulation level of heavy metals in plants by evaluating the percentage of heavy metals that is dissolved in water and in weak organic acids (KNO₃, EDTA).

Table 22: Heavy metals speciation in compost resulting from the 2nd composting trial [mg/kg]

Heavy Metals	Total	Dissolved in H ₂ O	Dissolved in KNO ₃	Dissolved in E.D.T.A	Total dissolved metals	Total dissolved [%]
Cd	0.5974	0.0048	0.0107	0.0189	0.0343	5.75
Cr	11.1931	0.0112	0.2200	0.0211	0.2523	2.25
Cu	54.1748	0.5046	0.4315	1.5749	2.5110	4.64
Ni	11.9650	0.3080	0.1042	0.2602	0.6723	5.62
Pb	20.0321	0.0335	0.0368	0.6310	0.7013	3.50
Zn	109.2423	0.6750	0.1255	3.7048	4.5053	4.12

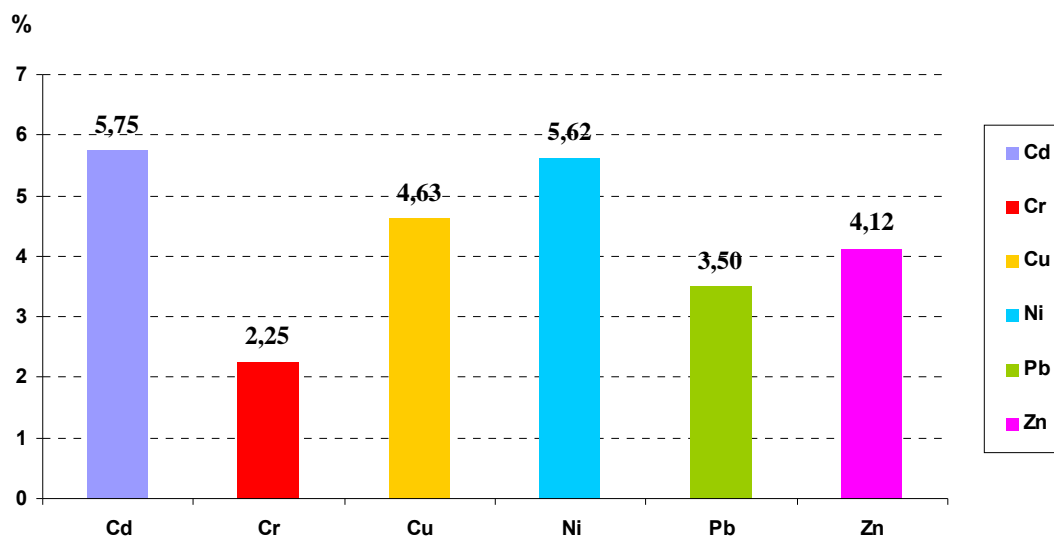


Figure 22: Percentages of total dissolved heavy metals in compost resulted from the 2nd composting trial

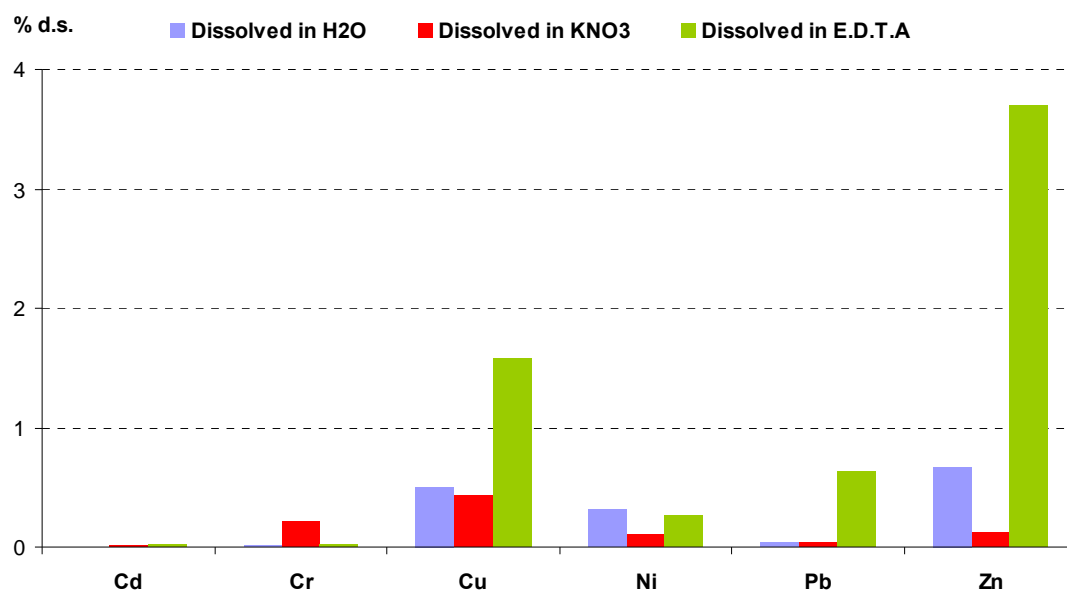


Figure 23: Heavy metals content [% d.s] that is dissolved in water and in weak organic acids in compost resulted from the 2nd composting trial

According to Table 22 and Figure 22 Ni and Cd have the highest percentages of total heavy metal dissolution, rated at 5.75% and 5.62% respectively followed by Cu and Zn at 4.63% and 4.12% respectively but in real values the concentrations is low rating from 0.0343mg/kg to 4.5053mg/kg. However with respect to heavy metal content that is dissolved in water, KNO₃ and EDTA (Table 22 and Figure 23), Zn, Cu and Pb were the heavy metals with the highest quantity of potential accumulation in plants. This in accordance to the cumulative research in Europe into the agronomic use of compost, according to which heavy metals tend to accumulate in soil and plants in the following order: Zn > Cu > Pb = Cd > Ni > Cr [59, 60].

Biological assays

To estimate the reduction of the pathogenic microorganisms, analyses have been performed to evaluate the density of total coliforms, faecal coliforms and helminth eggs prior and after the 2nd composting processes (Tables 18 and 23).

Table 23: Micro-biological parameters in compost produced in the 2nd composting trial

Micro-biological parameters	Compost
Total coliforms Log10 MPN/10g DS	0.90
Faecal coliforms Log10 MPN/10g DS	0.30
Helminth eggs /10g DS	< 1

During the 2nd composting trial, elevated temperatures ($>55^{\circ}\text{C}$) were obtained for approximately 5 consecutive days as shown in Figure 13 (5th to 9th day). Sufficient hygienized end product is obtained if a certain temperature level is maintained during composting resulting in the elimination of the pathogenic microorganisms [12]. Therefore it can be inferred that the 2st composting trial had been performed successfully with respect to the sanitization of biosolids such as primary sludge since the levels of pathogenic microorganisms detected at the end of the process were significantly lower than initially [12, 13, 14].

5.2.10. Preparing the system for the implementation of the next composting trial

After the removal and collection of the compost the electromechanical components of the system were monitored and maintained while the whole system was cleaned up thoroughly. The maintenance and monitoring of the system were carefully planned and carried out according to the instructions specified in the operation and maintenance manual of the manufacturer.

5.3. Composting using primary sludge, green waste, manure and zeolite – 3rd trial

In this section the experimental results obtained from the analysis of feedstock of the 3rd composting trial are shown. The feedstock includes primary sludge from El Jadida city, green waste, manure and zeolite as additive. Initially the feedstock was chosen and the composition of the raw material was determined. Next the operational conditions of the bioreactor were programmed taking into consideration the experience and results obtained in the previous composting trials in order to improve and optimize the overall process, such as hydration, aeration and temperature control. Composting samples were taken at regular time intervals from the substrate and leachates for analyses and evaluation of the process. Samples were also collected from the end product for detailed analyses such as physicochemical analyses and biological assays.

5.3.1. Sampling and analysis of the raw material

Table 24 presents the physicochemical characteristics of the raw materials that were used as feedstock for the operation of the 3rd composting trial.

Table 24: Physicochemical parameters of the raw material used in the 3rd composting trial

Parameter	Primary sludge	Cow Manure	Sugar beet leaves
Dry Solids (% d.s)	31.3	92.56	52.10
pH	6.5	9.2	7.1
Total C (% d.s)	12.6	45.43	61.50
Total N (% d.s)	1.65	2.65	3.48
C:N ratio	7.64	17.14	17.67
Total P, as P ₂ O ₅ (% d.s)	1.0715	0.5620	0.2605
K, as K ₂ O (% d.s)	0.8540	3.7545	3.5878
Ca as CaO (% d.s)	4.2507	6.9176	3.6205
Mg as MgO (% d.s)	2.0860	1.2601	1.5463
Cd (mg/Kg d.s)	1.1731	0.7858	0.1022
Cr (mg/Kg d.s)	21.6286	13.8386	0.8056
Cu (mg/Kg d.s)	175.3108	24.65726	13.414

Ni (mg/Kg d.s)	31.4635	16.0901	1.3201
Pb (mg/Kg d.s)	91.6719	2.17552	0.3830
Zn (mg/Kg d.s)	225.5545	45.4673	36.5834

According to Table 24 the primary sludge used in the 3rd composting trial acquired increased moisture content of 68.7% since the collected sludge did not undergo dewatering. On the other hand cow manure and sugar beet leaves had much lower water content of 7.44% and 47.9% respectively which can balance the level of the substrate's water level when mixed with primary sludge. The pH of primary sludge and sugar beet leaves was near neutral which is considered satisfactory for the development of composting processes while cow manure adds to the alkalinity since it acquires a pH value of 9.2. With respect to the total carbon content sugar beet leaves acquired the highest carbon content (61.50%) followed by cow manure and primary sludge with 45.43% and 12.6% respectively. The nitrogen content is high proportionally to the carbon content for all the raw materials leading to low carbon to nitrogen ratios of 7.64, 17.14 and 17.67 for the primary sludge, cow manure and sugar beet leaves respectively. Furthermore, the physical properties of sugar beet leaves make a good quality structuring agent for mixing with sewage sludge. As has been mentioned the macroelement content incorporated to the feedstock is crucial in determining the evolution of the composting process since those elements are used in the greatest quantities by plants and are the nutrients most often applied through commercial fertilisers. According to the characteristics of the raw material (Table 24) the NPK value, as well as the rest macroelement content, appear to be sufficient for the purposes of composting. With respect to the heavy metal content primary sludge does not have limiting heavy metal values for the composting process since it does not exceed the limits for sludge application in agricultural land as has been set by Directive 86/278/EEC (Table 3).. Although urban residues are generally emphasized by high heavy metal content in the case of primary sludge used in the 3rd composting trial, the concentration appears to be low in comparison to the limit values of heavy metal concentrations for sludge use for agricultural purposes.

Table 25 shows that the primary sludge used in the 3rd composting trial contains high number of pathogenic microorganisms which constitutes a health hazard and if sludge

is used for agricultural purposes without being submitted to a proper treatment process, such as composting, there is a strong risk of contamination [12, 13, 14, 15].

Table 25: Micro-biological parameters of primary sludge used in the 3rd composting trial

Micro-biological parameters	Primary sludge
Total coliforms Log10 MPN/10g DS	10.06
Faecal coliforms Log10 MPN/10g DS	7.44
Helminth eggs /10g DS	24.60

The zeolite that was used into the 3rd composting trial was clinoptinolite originated from Evros region, Greece. The zeolite was converted into ionic form by adding it into NaCl solution. Zeolite was then undergo multiple rinsing using distilled water, desiccation and storage into a NaCl saturated solution for two weeks. Table 26 presents the zeolite content in Si, Al, Fe, Ca, Na, K, Mg, Mn and water content.

Table 26: The characteristics of zeolite used in the 3rd composting trial

Parameters [% w/w]	Zeolite
Si	60.715
Al	12.035
Fe (Fe ₂ O ₃)	1.398
Ca (CaO)	1.505
Na (Na ₂ O)	6.975
K (K ₂ O)	0.490
Mg (MgO)	0.129
Mn (MnO)	0.049
water content	14.455

Clinoptinolite is characterised by its ability to exchange sodium/potassium ions with ammonium ions and heavy metals ions. Clinoptinolite is very selective to ammonium ions and thus prevents it from escaping to atmosphere as ammonia gases. During composting ammonia can be taken by zeolite in exchange of other mobile ions. In a similar mechanism heavy metals can be trapped by clinoptinolite. In this way heavy metals are not so mobile and they can stay on the zeolite structure instead of moving

into the soil and thus to the plants. Clinoptinolite is a good structuring material allowing, during composting, the substrate to be better aerated .It has also great water capacity, a characteristic that is very important when compost is applied to the fields.

5.3.2. Composition of the composting raw material

The feedstock material of the 3rd composting trial comprised of primary sludge from El Jadida city, cow manure, sugar beet leaves and zeolite as additive. The quantities of each material used are given in Table 27.

Table 27: Composition of the mix used for composting in the 3rd composting trial

Raw material	Kg w.w.
Primary sludge	700
Cow manure	180
Sugar beet leaves	450
Zeolite	50

The amount of zeolite to be used was calculated taking into consideration the previous trials especially the ammonium and heavy metals concentrations. As mentioned above clinoptinolite is very selective for ammonium and heavy metal ions.

5.3.3. Pretreatment and preparation of the composting raw material

The sugar beet leaves were shredded to size 2 to 4 cm using a commercial shredder type MTD 118E prior its supply into the bioreactor. After the green material had been shredded it was then weighted and packed ready to be used. The primary sludge, the cow manure and the zeolite were also weighted and packed ready to be used. The weighted and packed raw materials were then mixed manually using shovels and ready to be fed into the bioreactor.

5.3.4. Feeding the in-vessel bioreactor

The feeding of the bioreactor was performed using the mixture prepared as indicated in paragraph 5.3.3 via the conveyor belt in which the feedstock was loaded manually using shovels.

5.3.5. Composting process control

Composting process control involves all the actions that had been planned and carried out with respect to the operational conditions of the in-vessel bioreactor.

5.3.5.1. Programming the plc of the in-vessel bioreactor

The operation of the agitation, hydration and aeration systems was performed by the PLC automatic control system. These operations can also be performed manually. The programming of each operation during the 3rd composting trial is given below:

Programming the frequency and duration of the substrate's agitation:

For the first 10 days of the process the motor was set to operate 4 times per day (every 6 hours) for 5 minutes each time. For the next 7 days the motor was programmed to operate 3 times per day (every 8 hours) for 5 minutes each time and for the rest days of the process it operated 2 times per day (every 12 hours) for 5 minutes each time.

Programming the frequency and duration of the substrate's aeration:

For the first 10 days of the process the fan was set to operate 4 times per day (every 6 hours) for 5 minutes each time. For the next 7 days the fan was programmed to operate 3 times per day (every 8 hours) for 5 minutes each time and for the rest days of the process it operated 2 times per day (every 12 hours) for 5 minutes each time.

Programming the frequency and duration of the substrate's hydration:

The hydration system operated manually during the 3rd composting trial although it could be programmed to operate automatically. The substrate was subjected to hydration whenever it was considered appropriate according to its moisture content that had been recorded from the daily measurements. The duration of the hydration

operation as well as the water flow were also adjusted according to the moisture content of the substrate that had been recorded from the daily measurements

5.3.5.2. Measuring the temperature of the substrate

Compost heat is produced as a by-product of the microbial breakdown of organic material. The heat production depends on the moisture content, aeration, and C:N ratio as well as on the volume of the substrate. Figure 24 presents the temperature evolution in the 3rd composting trial as has been recorded.

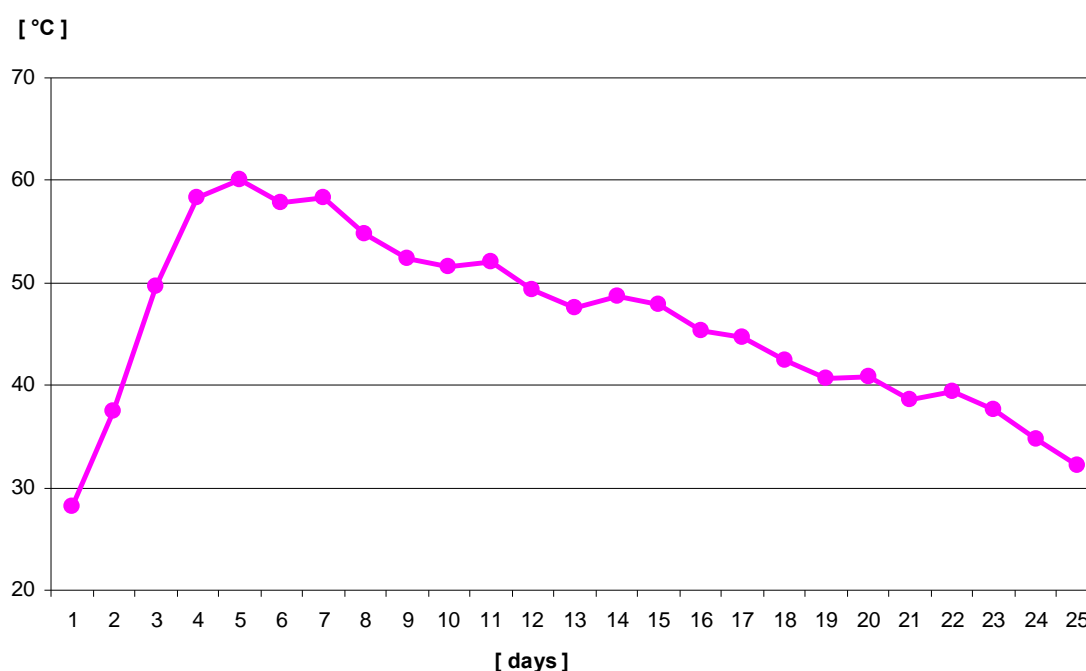


Figure 24: Temperature profile during the 3rd composting trial

According to Figure 24 the substrate passed from an initial mesophylic phase (<40°C) to a thermophylic stage after the 2nd day of the initiation of the composting process. The rate of processing in a biochemical system is directly proportional to the increase of temperature. Microbial activity during the 3rd composting process released large amounts of heat which accumulated due to the initial fast decomposition of the soluble, readily degradable compounds (present mainly at the primary sludge compounds and cow manure) and due to the self-insulating qualities of the compost material the temperature rose rapidly and to reached to a maximum of 60.1°C on the 5th day. Elevated temperatures (>50°C) are maintained within the bioreactor for 9 continuous days (3rd to 11th day) which is sufficient time for the sterilisation of the substrate from potential pathogenic microorganisms that exist in the composting

material. Only after all substrate, capable of promoting microbial activity, had been used up did the temperature begin to fall to reach a second mesophylic phase on the 21st day of the process while the maturation process took place during this last stage of composting.

5.3.5.3. Measuring the moisture of the substrate

A moisture content of 40-60% is generally considered optimum for composting. Microbially induced decomposition occurs most rapidly in the thin liquid films found on the surfaces of the organic particles. Whereas too little moisture (<30%) inhibits bacterial activity, too much moisture (>65%) results in slow decomposition, odour production in anaerobic pockets, and nutrient leaching. Figure 25 presents the moisture evolution in the 3rd composting trial as has been recorded.

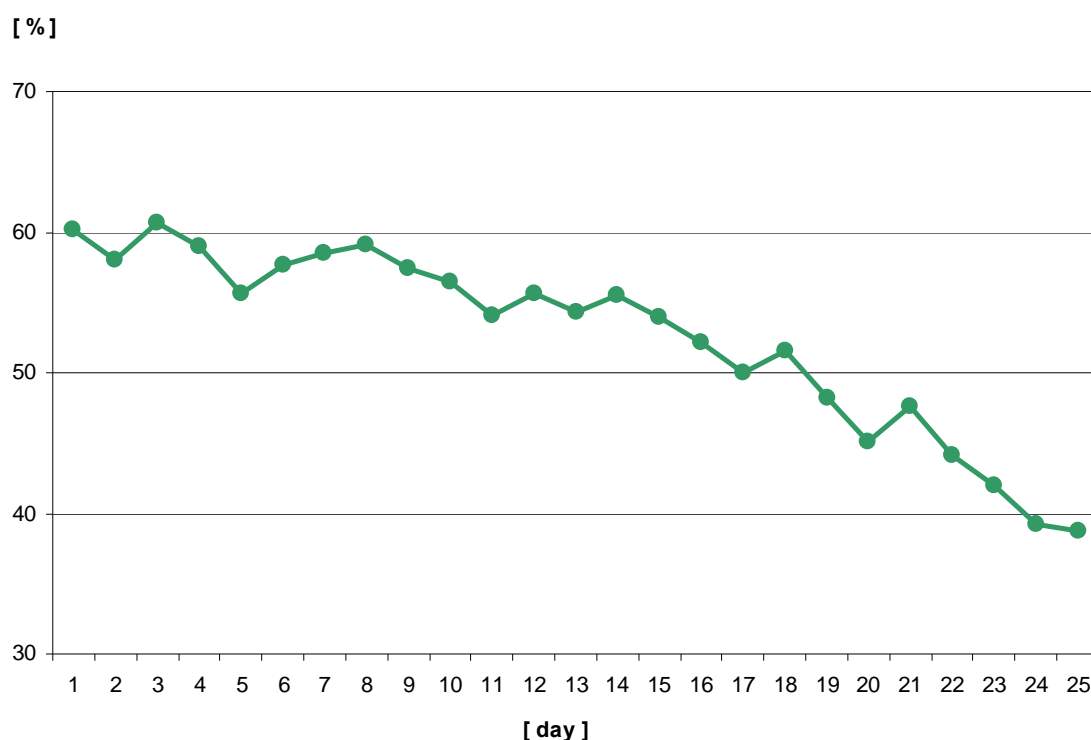


Figure 25: Moisture content profile during the 3rd composting trial

From the results obtained, the initial substrate had a moisture content of 60.2% which is within the optimal range as stated above. The presence of readily degradable organic matter in conjunction to sufficient substrate's agitation and oxygen supply enhanced the metabolic activities microbes and caused the temperature to rise, leading to moisture content reduction as composting was developing. The moisture content was sustained at optimal levels around 40% to 60% throughout the duration of the 3rd

composting trial due to the manual hydration of the substrate and its sufficient agitation and aeration. At the final stage of composting the water quantity was decreased reaching 38.8% at the final day of the process.

5.3.5.4. Measuring the oxygen content of the substrate

As has been mentioned the production of heat and the need for oxygen are greatest at the early stages due to the readily degradable components and then steadily decrease. Composting process takes place even in low oxygen content (5%) however, oxygen concentrations greater than 10% are consider optimal for maintaining aerobic conditions and thus sustaining aerobic microbial activity. Figure 26 presents the moisture evolution during the 3rd composting trial.

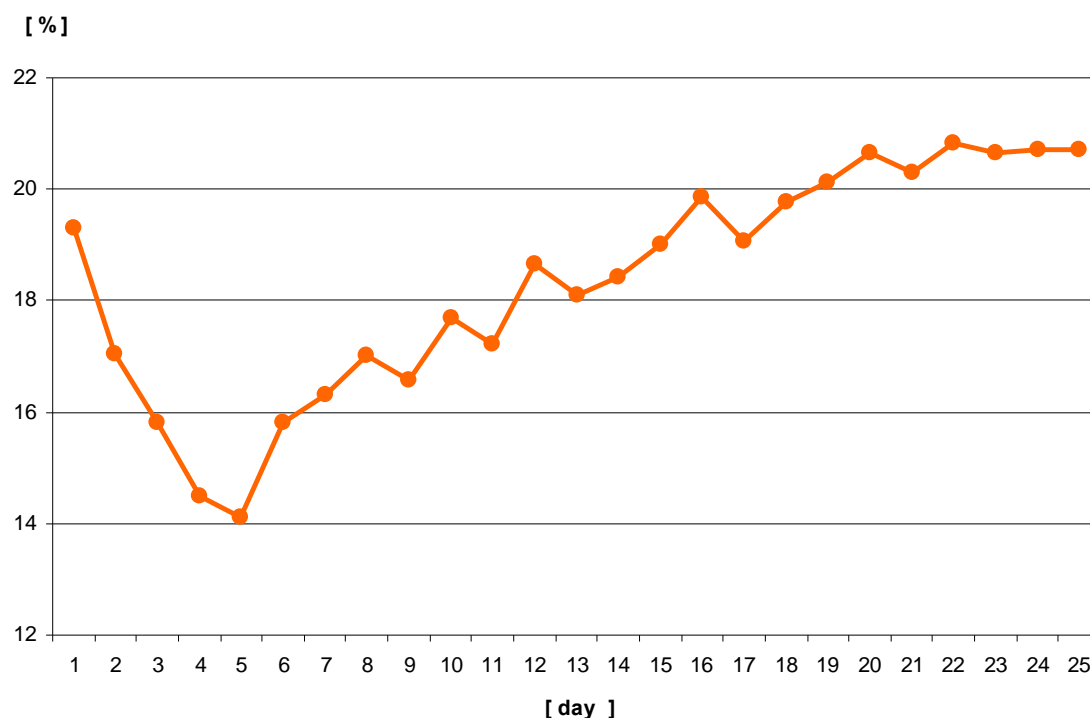


Figure 26: Oxygen profile during the 3rd composting trial

According to Figure 26 the oxygen concentration of the substrate was maintained higher than 14.0% throughout the duration of the 3rd composting trial. The lowest oxygen concentration values were recorded during elevated temperatures (3rd to 11th day) due to the high microbial activity of aerobic microorganisms which consume oxygen for the decomposition of the organic matter. The increased oxygen level and the incremental tendency of oxygen concentration during composting indicate that the agitation and thus the aeration of the substrate were sufficient in promoting aerobic

conditions in the bioreactor. At the latest stage of the composting process (19th day onwards) the level of oxygen maintained higher than 20.0% indicative of the low rate of organic matter decomposition since the concentration of oxygen nearly reached that of the atmosphere (21%).

5.3.5.5. Reprogramming the plc of the in-vessel bioreactor

The plc of the in-vessel bioreactor was not reprogrammed during the 3rd composting trial

5.3.5.6. Monitoring and maintenance during composting

Throughout the duration of the composting process the electromechanical equipment and the rest of the bioreactor's components and infrastructure were being monitored daily in order to maintain their operation status. The regular maintenance of all the electromechanical equipment of the system was performed according to the suggestions of the manufacturer (e.g. lubrication, cleaning the filter of the ventilation system, cleaning the head of the piping system, leachate removal). In addition, daily cleaning of the floor and surface of the bioreactor was performed in order to obtain high hygiene level for the personnel.

5.3.5.7. Disruption and startup of the bioractor operation

No disruptions of the bioreactor's operation occurred during the 3rd composting trial which lasted for 25 days.

5.3.5.8. Restoration of malfunction and maintenance of the bioreactor

No malfunctions occurred during the 3rd composting trial

5.3.6. Sampling and analysis of the substrate

Substrate samples were taken every 4 to 6 days of the composting process and the examined parameters included the pH, total carbon, total nitrogen, nitrates ($\text{NO}_3^- - \text{N}$) and ammonium nitrogen ($\text{NH}_4^+ - \text{N}$).

5.3.6.1. pH

Figure 27 presents the evolution of the pH values during the 3rd composting trial. By examining the graph it appears that the changes in pH values follow the same pattern as the pH values of the previous composting trials.

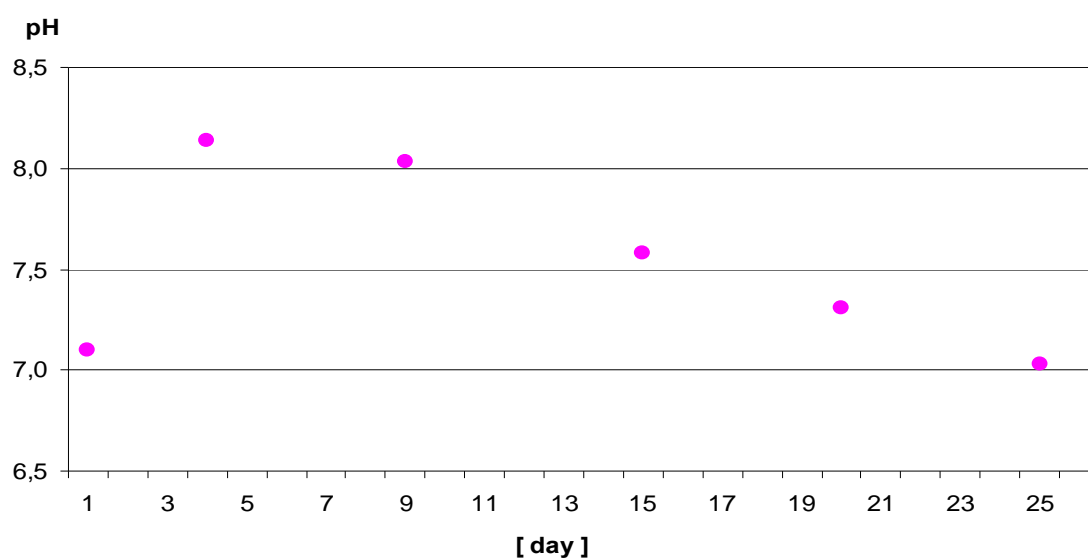


Figure 27: Changes in pH values during the 3rd composting trial

The pH increased during the initial stage of composting (7.10 to 8.14 on the 1st and 5th day respectively) probably due to the activity of proteolytic bacteria which release ammonia from the degradation of the organic matter. Then through ammonia volatilization and oxidation to nitrates the ammonia content decreased and the pH dropped from 8.14 to 7.03 at the final day (25th) of the process.

5.3.6.2. Total Carbon

The changes occurred in the total carbon content during the 3rd composting trial resemble to the changes of total carbon content during the 2nd composting trial. Figure 28 presents the evolution of the carbon content during the 3rd composting trial.

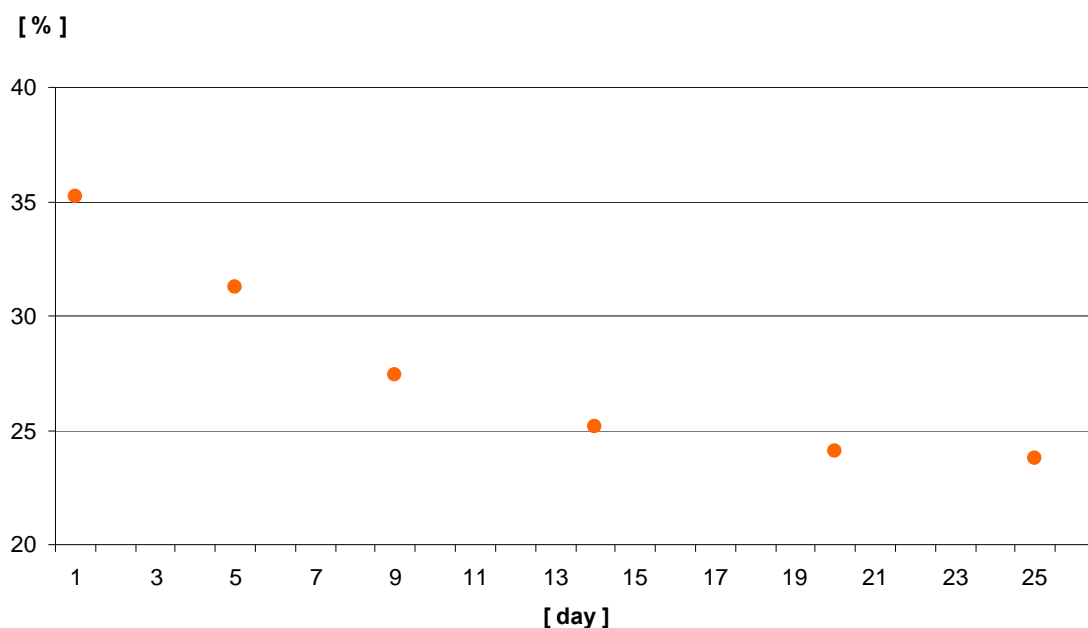


Figure 28: Changes in total carbon content [%] during the 3rd composting trial

At the early stages of the process carbon loss occurred due to the bio-oxidation of the labile organic matter. That loss accounted to 7.81% on the 9th day since the initiation of the process. As composting was developing the rate at which carbon was consumed decreased due to the depletion of the available organic compounds capable of promoting intensive microbial activity and thus the carbon loss from the 9th day onwards accounted to 3.63%. This is indicative of the fact that the highest carbon transformation/biooxidation occurs mainly when intensive microbial activity takes place. At the end of the process the carbon has been reduced to 23.80% achieving an overall 11.44% decrease to the carbon content of the substrate in comparison to the initial mixture. In regard with the carbon content the end product resulting from the 3rd composting trial is of good quality [19].

5.3.6.3. Total Nitrogen

Figure 29 presents the total nitrogen content evolution during the 3rd composting trial.

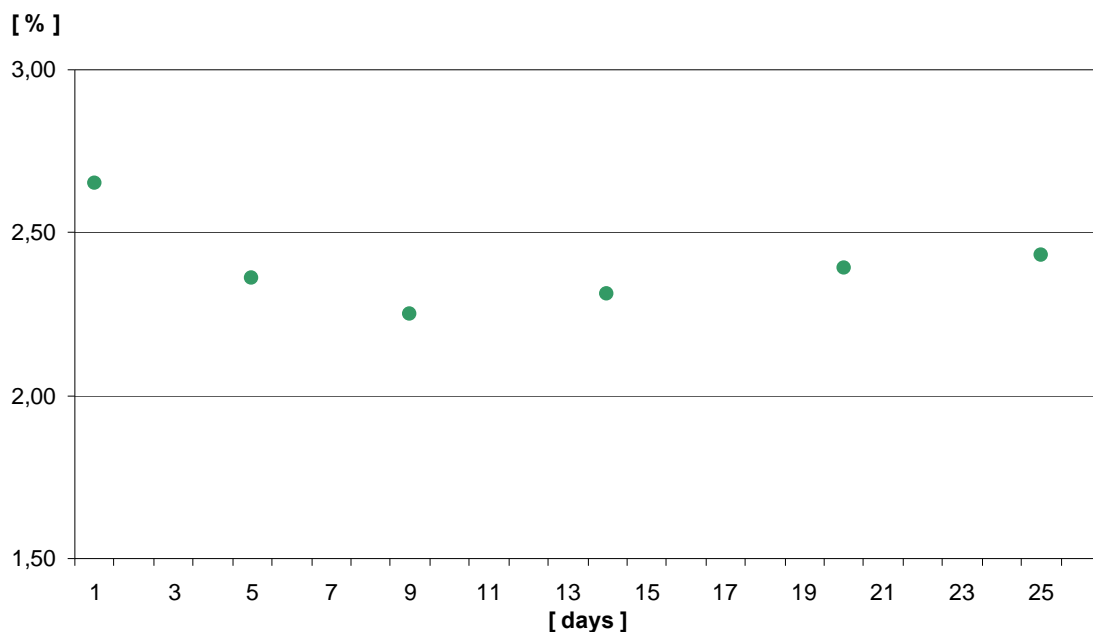


Figure 29: Changes in total nitrogen content [%] during the 3rd composting trial

During the first phase of the process the combination of low C:N ratio, high ammonium concentration, high temperatures, and raised pH values led to increased ammonia losses. The ammonia losses in conjunction to the initial rapid carbon biooxidation explain the decrease of nitrogen during the first 9 days of the process [65]. The level of nitrogen losses through ammonia volatilization varies according to the raw material that is used for composting and can reach up to 50% of the initial total nitrogen [22, 23]. However the use of zeolite as amendment prohibits the loss of ammonia through volatilization by trapping ammonium [66]. From the 9th day onwards the nitrogen content presented an increase which is mainly attributed to the activity of nitrogen-fixing bacteria (azotobacters) [21]. The nitrogen content at the end of the 3rd composting trial on the 25th day was 2.43% which is lower than the initial nitrogen content (2.65%).

5.3.6.4. Ammonium Nitrogen (NH_4^+ - N)

Figure 30 presents the changes of ammonium content during the 3rd composting trial. According to Figure 30, the ammonium evolution in the 3rd composting trial follows the same pattern as in the previous trials.

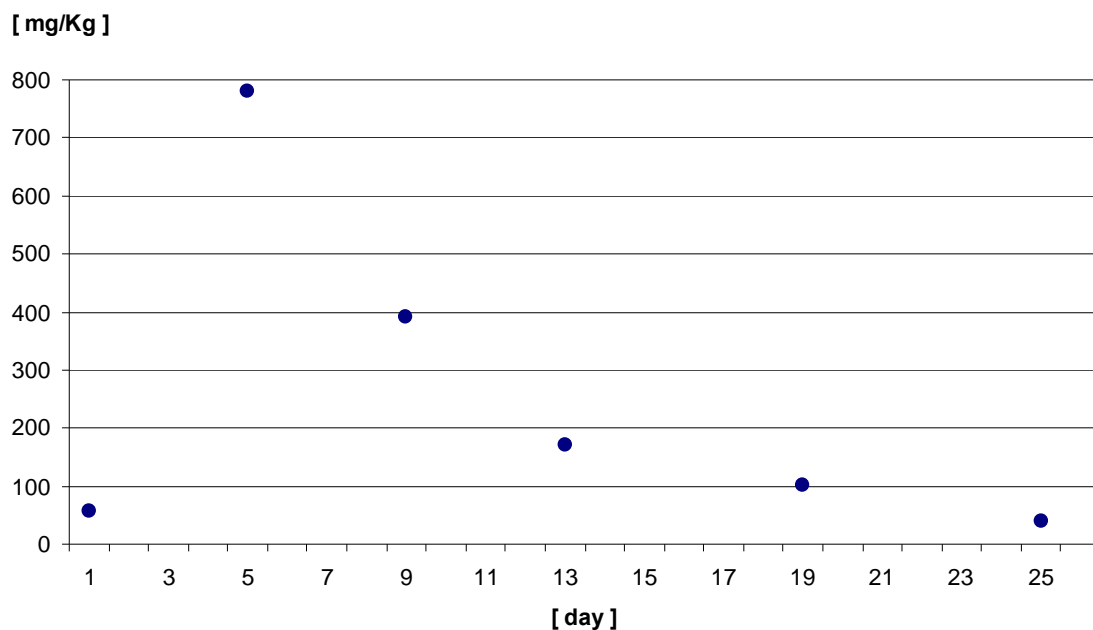


Figure 30: Changes in ammonium concentration [mg/kg] during the 3rd composting trial

Ammonium was formed during the first phase of organic matter degradation reaching to a maximum of 780.42mg/kg on the 5th day of composting. The maximum concentration achieved in the 3rd composting trial was lower than that of the previous trials which is attributed to the use of clinoptinolite by removing significant quantity of ammonium. Clinoptinolite represents high selectivity to ammonium even higher than the selectivity to heavy metals. Since after the 5th day of the composting process the concentration of ammonium was reduced through volatilization¹⁰ and oxidation to nitrates by the action of nitrifying bacteria and by the end of the process on the 25th day ammonium has reached 38.65mg/kg.

5.3.6.5. Nitrates (NO₃ - N)

Besides ammonium, nitrates concentration also changes during the 3rd composting trial in the same manner as in the previous composting trials. Figure 31 presents the evolution of nitrates content during the 3rd composting trial.

¹⁰ Mainly due to elevated temperature values, increased pH, agitation and aeration

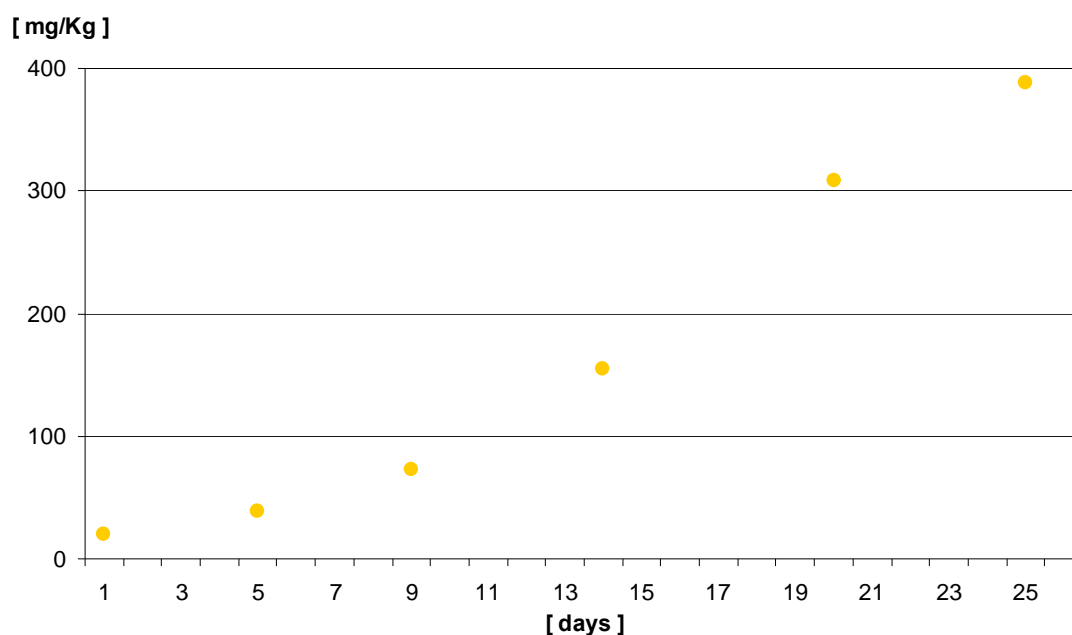


Figure 31: Changes in nitrates concentration during the 3rd composting trial

Heterotrophic nitrification was present at the early phases of the process since the production of nitrates at elevated temperatures and increased ammonium concentration was almost exclusively the work of heterotrophic-nitrifiers reaching 38.74mg/kg and 72.58mg/kg on the 5th day and 9th day respectively [34, 67, 68]. Autotrophic nitrification was detected at a later stage of composting since this type of nitrification is markedly affected by high temperature as well as excessive quantities of ammonia which barrier the growth of autotrophic-nitrifiers (e.g. nitrobacteria). Thus on the 14th day onwards the nitrates concentration increased and reached up to 388.12mg/kg at the end of the process. It must emphasized that the level of nitrates in the substrate resulting from the 3rd trial is much lower compared to the previous trials. This is explained by the fact that the level of ammonium concentration in the substrate is reduced significantly due to the presence of zeolite so there is less available ammonium to be converted to nitrates.

5.3.6.6. C:N Ratio

As has been mentioned raw materials mixed to provide a C:N ratio of 25:1 to 30:1 are generally accepted as ideal for active composting, although ratios from 20:1 up to 40:1 can give good composting results. The initial raw composted material comprised of primary sludge, cow manure and sugar beet leaves (Table 27) which acquire low

C:N ratios as has been shown in Table 24. Although it was expected that the substrate would initially acquire a low C:N ratio it was considered appropriate to examine whether composting process is effective for these types of waste since their low cost, their availability and the amount of each material type were suitable for the purposes of the project. Figure 32 presents the C:N ratio evolution during the 3rd composting trial.

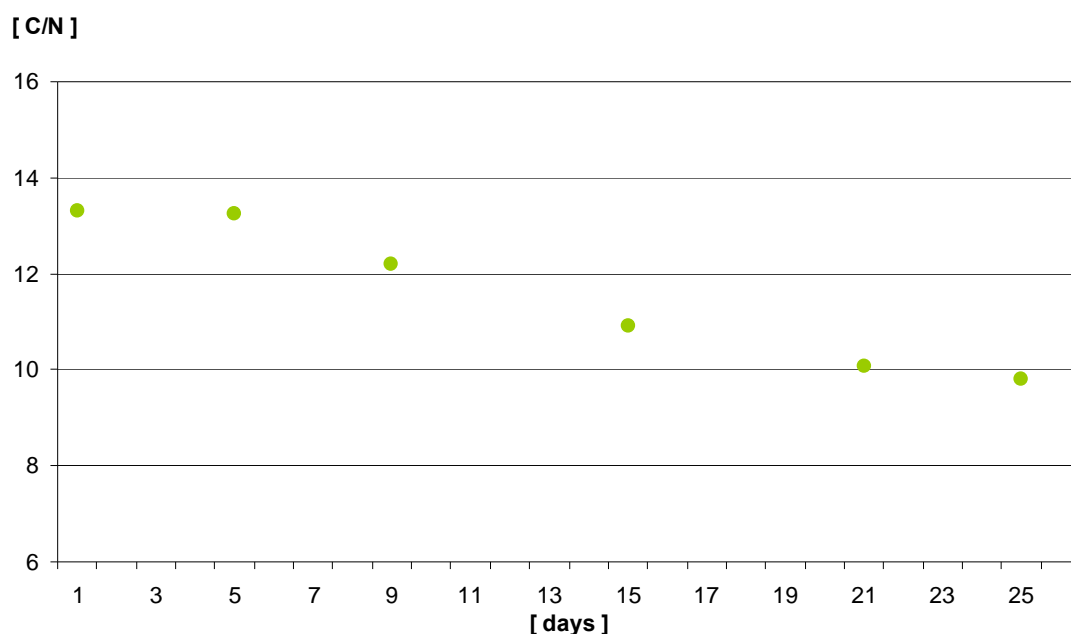


Figure 32: Changes in C:N ratio during the 3rd composting trial

According to Figure 32 the initial C:N ratio was 13.30 and remained constant during the first five days of the process due to the simultaneous losses of carbon through biooxidation to CO₂ and the losses of nitrogen through ammonia volatilization and ammonium escape through leachates. From the 5th day onwards the C:N ratio decreased due to the continuous carbon loss and the increase of nitrogen content by the activity of nitrogen-fixing bacteria (azotobacters). At the end of the process the C:N ration has been decreased to 9.79 which is qualified as of good quality compost that can be applied in agricultural land since composts acquiring a C:N ratio lower than 20 do not interfere with plant nutrition in the soil [2, 15] while too low C:N ratio can cause ammonium toxicity [44, 45].

5.3.7. Sampling and analysis of the substrate's leachate

Table 28 presents the leachate parameters examined during the 3rd composting trial. The parameters examined are pH, BOD₅, ammonium, nitrates and heavy metals.

Table 28: Characteristics of leachates resulting from the 3rd composting trial

Parameter/Time	3 rd day	7 th day	14 th day	20 th day
pH	7.8	8.0	7.6	7.2
BOD (mg/L)	56905	35793	21805	11554
NH ⁴⁺ - N (mg/L)	50.47	412.35	164.70	92.57
NO ³⁻ - N (mg/L)	24.72	63.49	140.58	259.90
Cd (mg/L)	0.00219	0.00138	0.00069	0.00058
Cr (mg/L)	0.02020	0.01608	0.14736	0.01125
Cu (mg/L)	0.19886	0.20780	0.19683	0.20152
Ni (mg/L)	0.04477	0.03307	0.03137	0.03672
Pb (mg/L)	0.05814	0.04172	0.04508	0.05067
Zn (mg/L)	0.29772	0.30226	0.32347	0.35740

The pH in leachates increased slightly during the initial stage reaching to a maximum of 8.0 on the 7th day starting from 7.8 and then it was gradually reduced acquiring a near neutral pH of 7.2. The initial sample of leachates derived from the 3rd composting trial acquired an increased initial BOD₅ value of 56.9g/L on the 3rd day. As the process kept developing the need for oxygen in leachate decreased and thus the BOD₅ values declined and reached on the 20th day the value of 11.6g/L which is approximately 1/5 of the initial BOD₅. Leachates acquired initially a light dark colour which was gradually becoming darker as composting was developing. This is indicative of the presents of humic/fulvic and other complex compounds. Ammonium and nitrates in leachates followed the same pattern as the ammonium and nitrates of the substrate indicating that the composting process was well monitored since the conditions were in favor of nitrification. Ammonium concentration in leachate showed a steep increase during the first days of composting from 50.47mg/L on the 3rd day to a maximum of 412.35mg/L on the 7th day. From that day onwards ammonium concentration was reduced and on the 20th day ammonium concentration was 92.57mg/L. It must be emphasized that the level of ammonium in leachates produced in the 3rd composting trial was much lower than in the previous trials. This is due to the fact the presence of zeolite in the substrate removed ammonium from the

leachates through the ion exchange process. Nitrates in leachates presented an increase throughout the duration of the 3rd trial. On the 3rd day their concentration was limited at 24.72mg/L while on the 20th day it had increased approximately 10 times reaching at 259.9mg/L. The level of nitrates in leachates resulting from the 3rd trial was the lowest compared to the previous trials. This is explained by the fact that the level of ammonium concentration was much lower in the 3rd trial than in previous ones due to the presence of zeolite so there was less available ammonium to be converted to nitrates. Table 28 shows also that heavy metals such as Cd, Cr, Cu, Ni, Pb and Zn were present in leachates throughout the process. The metal concentrations were much below the indicative values for wastewater disposal as shown in Table 7. This is so due to the presence of zeolite which has the ability to remove metal ions.

Although composting can be assessed for sludge treatment and reuse through land application, leachate remains an environmental problem since the treatment of solid waste through composting results in transferring the problem from solid to liquid phase. Leachate must be treated accordingly in order to be disposed to a water recipient based on the suggested standards shown in Table 7. However Directive 91/271/EEC seeks to harmonise measures relating to the treatment of wastewater at Community level in order to protect the environment from any adverse effects due to discharge of such waters. According to BOD₅, ammonium and nitrates values obtained for the leachates of the 3rd composting trial it can be concluded that leachates had a heavily polluted organic and inorganic load which needs to be treated before its final disposal. The quantity of leachate production during the 3rd trial was limited and stored to an impermeable reservoir for sufficient period of time to be stabilized at lower values through aeration.

5.3.8. Removal and collection of the composting end product

The removal and collection of compost was performed on the 25th day of the composting process through the removal portals that are situated at the bottom part of the bioreactor. To facilitate the discharge of the end product the agitation system was operated for small intervals. The compost was collected manually using shovels and it was spreaded in an open area outside the building where the bioreactor is installed where it remained for five days to further reduce its moisture content (during that time

the end product was being regularly agitated manually). Finally the compost was stored in order to be used to the open field experiments.

5.3.9. Sampling and analysis of the produced compost

The compost physicochemical parameters examined are given in Table 29.

Table 29: Physicochemical parameters of the compost produced in the 3rd composting trial

Parameter	Compost
Dry Solids (% d.s.)	61.2
pH	7.03
Total Carbon (% d.s.)	23.80
Total Nitrogen (% d.s.)	2.43
C:N ratio	9.79
NO ₃ ⁻ - N (mg/Kg.d.s)	388.12
NH ₄ ⁺ - N (mg/Kg.d.s)	38.65
Total P as P ₂ O ₅ (% d.s.)	0.79322
K as K ₂ O (% d.s.)	3.21070
Ca as CaO (% d.s.)	5.74819
Mg as MgO (% d.s.)	2.02320
Cd (mg/Kg d.s.)	0.72949
Cr (mg/Kg d.s.)	13.51757
Cu (mg/Kg d.s.)	82.47363
Ni (mg/Kg d.s.)	17.38393
Pb (mg/Kg d.s.)	38.15530
Zn (mg/Kg d.s.)	118.22976

The water content of compost produced in the 3rd trial was 38.8% which indicates that no intensive biological process can take place in such low moisture level. In addition the ideal level of water content at the end product ranges from 35% to 45% and in any case it should not be lower the 25% since composts acquiring such low moisture content cannot be easily incorporated into the soil [2, 3, 43]. The pH was neutral (7.03) indicating a good quality compost and within the suggested range of 6 to 8.5

[2, 3, 4, 5, 6] for the development of bacteria and fungi [69]. The total carbon was subjected to an overall 11.44% reduction throughout the 3rd composting process while the remaining carbon content at the end of the process was 23.08% which is considered to be a good value [20].

The appearance of significant quantities of nitrates at the end of the 3rd composting trial (388.12mg/kg) in conjunction to the low concentration of ammonium (38.65mg/kg) showed that oxidizing factors persisted during the process while the ammonium nitrates ratio of 0.1 indicates that the produced compost is mature since ratios lower than 0.5 are indicative of fully mature composts [72].

Maintaining C:N ratio after composting is crucial in determining the agronomical value of finished compost. As has been mentioned compost with high C:N (>20) can cause nitrogen immobilization upon amendment to soil while too low C:N ratio can cause ammonium toxicity [2,15, 44, 45]. Studies suggest various ideal C:N ratios from lower than 12 to lower than 25 [46, 47, 48]. This variation is possibly attributed to the different initial feedstock [49]. Therefore the 9.79 ratio obtained in the 3rd composting trial is considered to be satisfactory for plant growth. The macro-elements are chemical elements consumed in the greatest quantities by plants and they are needed in relatively large quantities. Table 9 presented indicative values of chemical nutrients content in compost based on international literature. As can be seen in Table 29 the macronutrients content of compost produced in the 3rd trial were sufficiently high and in accordance to literature review suggesting that compost would provide the required quantities of nutrients to plants. On the other hand the trace elements or heavy metals are needed by plants in very small quantities and increased concentrations of these minerals in compost may be damaging for plant growth. The heavy metals concentration of the produced compost was within the range of EU states heavy metal limits (Table 11) and complies even with the stringent German standards for maximum heavy metals concentration for intensive compost (Table 10) with the exception of Cu¹¹. In addition, the 3rd compost was in accordance with the environmental quality classes with respect to the heavy metal content and rated as 1st class compost (Table 13).

¹¹ German standards Cu<50mg/kg, 3rd compost = 82.5mg/kg

Heavy metals speciation

Table 30 and Figures 33 and 34 present the potential heavy metal accumulation in plants which is shown as the percentage of heavy metals that is dissolved in water and in weak organic acids (KNO₃ and EDTA). As has been stated in the 3rd composting trial clinoptinolite was used as additive. The clinoptinolite used in the composting trial was quite coarse (about 5mm) so that it could be removed from the compost samples in which the heavy metal analysis was carried out.

Table 30: Heavy metals speciation in compost resulting from the 3rd composting trial [mg/kg]

Heavy Metals	Total	Dissolved in H ₂ O	Dissolved in KNO ₃	Dissolved in E.D.T.A	Total dissolved metals	Total dissolved [%]
Cd	0.72949	N.D.	N.D.	N.D.	0.00000	0.000
Cr	13.51757	0.00019	0.00534	0.00488	0.01041	0.077
Cu	82.47363	0.00079	0.00420	0.00022	0.00521	0.006
Ni	17.38393	0.00016	0.00098	0.00143	0.00257	0.015
Pb	38.15530	0.00007	0.00152	0.00215	0.00374	0.010
Zn	118.22976	0.00111	0.02171	0.02988	0.05270	0.045

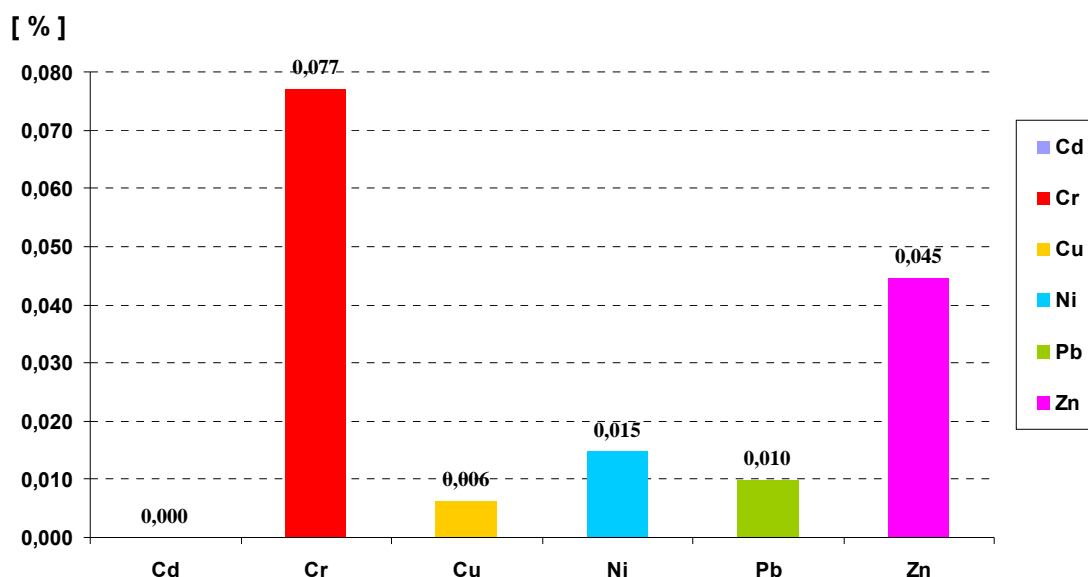


Figure 33: Percentages of total dissolved heavy metals in compost resulted from the 3rd composting trial

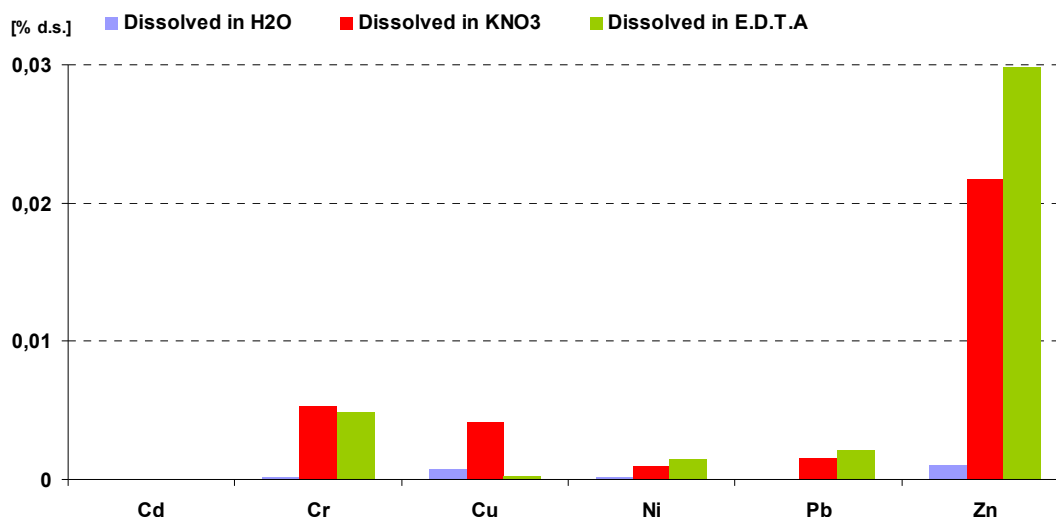


Figure 34: Heavy metals content [% d.s] that is dissolved in water and in weak organic acids in compost resulted from the 3rd composting trial

The total dissolved level of all heavy metals was less than 0.08% and in comparison to the previous composting trials¹² that level was very small indicating that clinoptinolite removed almost all the ion exchangeable forms of all heavy metals whereas the remaining heavy metal quantities were in an inert form which is innocuous for compost application in agricultural land. The selectivity of clinoptinolite in removing heavy metals had the following order $Cd > Cu > Pb > Ni > Zn \geq Cr$ [70].

Biological assays

For compost to be freely used, the end-product must have a very low concentration in pathogens. The primary method for controlling pathogens is to expose them to elevated temperatures for an extended period of time [71]. The density of total coliforms, faecal coliforms and helminth eggs in compost resulting from the 4th trial is shown in Table 31.

Table 31: Micro-biological parameters of compost produced in the 3rd composting trial

Micro-biological parameters	Compost
Total coliforms Log10 MPN/10g DS	1.60
Faecal coliforms Log10 MPN/10g DS	0.70
Helminth eggs /10g DS	<1

¹² 1st composting trial Figures 11,12 and Table 14, 2nd composting trial Figures 23,24 and Table 22

The elevated temperatures ($>55^{\circ}\text{C}$) obtained for approximately 5 consecutive days, as shown in Figure 24 (4th to 8th day), had an influence on the presence of pathogenic microorganisms and it seems that the temperature reached during the composting process was responsible for the elimination of the pathogenic microorganisms as shown in Table 31 in comparison to Table 25 [12]. Therefore it can be inferred that the thermophilic duration was long enough for the sanitization of the compost

5.3.10. Preparing the system for the implementation of the next composting trial

After the removal and collection of the compost the electromechanical components of the system were monitored and checked while the whole system was cleaned up thoroughly. The maintenance and monitoring of the system were carefully planned and carried out according to the instructions specified in the operation and maintenance manual of the manufacturer.

5.4. Composting using secondary sludge and perlite – 4th trial

In this section the experimental results obtained from the analysis of feedstock of the 4th composting trial are shown. The feedstock included secondary sludge from El Jadida city and perlite as additive. Initially the feedstock was chosen and the composition of the raw material was determined. Next the operational conditions of the bioreactor were programmed taking into consideration the experience and results obtained in the previous composting trials in order to improve and optimize the overall process, such as hydration, aeration and temperature control. Composting samples were taken at regular time intervals from the substrate and leachates for analyses and evaluation of the process. Samples were also collected from the end product for detailed analyses such as physicochemical analyses and biological assays.

5.4.1. Sampling and analysis of the raw material

Table 32 presents the physicochemical characteristics of the raw materials that were used as feedstock for the operation of the 4th composting trial.

Table 32: Physicochemical parameters of the raw material used in the 4th composting trial

Parameter	Secondary sludge
Dry Solids (% d.s)	21.45
pH	5.7
Total C (% d.s)	46.32
Total N (% d.s)	5.74
C:N ratio	8.07
Total P, as P ₂ O ₅ (% d.s)	3.68
K, as K ₂ O (% d.s)	0.46
Ca as CaO (% of d.s)	4.13
Mg as MgO (% of d.s)	0.82
Cd (mg/Kg d.s)	0.4820
Cr (mg/Kg d.s)	12.9371
Cu (mg/Kg d.s)	98.3961
Ni (mg/Kg d.s)	9.7721
Pb (mg/Kg d.s)	21.9202

Zn (mg/Kg d.s)	593.7208
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According to Table 32 the secondary sludge acquired increased moisture content of 78.55% since the collected sludge did not undergo dewatering. With respect to the total carbon content secondary sludge acquired increased total carbon of 46.32% . The nitrogen content of sludge was high proportionally to its carbon content, leading to low carbon to nitrogen ratios of 8.07. The macro-element content appeared to be sufficient for the promotion of the microbial activity and thus for the development of the composting process. Finally, the heavy metal concentration of secondary sludge was in accordance with the limits for sludge application in agricultural land as has been set by Directive 86/278/EEC (Table 3) and therefore it was considered that sludge would not inhibit composting process.

Table 33 shows that the secondary sludge used in the 4th composting trial contains high number of pathogenic microorganisms which constitutes a health hazard. Their accumulation in sludge occurs either by direct settling¹³ or by adsorption on suspended matter such as activated sludge flocs¹⁴ [54].

Table 33: Micro-biological parameters of secondary sludge used in the 4th composting trial

Micro-biological parameters	Secondary sludge
Total coliforms Log10 MPN/10g DS	10.30
Faecal coliforms Log10 MPN/10g DS	8.70
Helminth eggs /10g DS	69.00

The usual types of pathogens introduced in wastewaters and consequently in sludge consist of bacteria, viruses, protozoa, nematodes and fungi. These attack the human immune system causing diseases of the gastrointestinal tract such as typhoid, paratyphoid fever, dysentery, diarrhoea and cholera [54]. Therefore if sludge is used for agricultural purposes without being submitted to a proper treatment process, such as composting, there is a strong risk of contamination [12-15].

¹³ mainly eggs, cysts and protozoa that have sufficient density

¹⁴ bacteria and viruses

5.4.2. Composition of the composting raw material

The feedstock material of the 4th composting trial comprised of secondary sludge from a food industry and perlite which was used as additive. The quantities of each material used are given in Table 34.

Table 34: Composition of the mix used for composting in the 4th composting trial

Raw material	Quantity
Secondary sludge	1500 Kg
Perlite	500 L

As has been stated the secondary sludge had increased water content due to the fact that there was not a dewatering installation near the region where secondary sludge was produced. Therefore composting secondary sludge individually was not considered appropriate and the addition of perlite would modify the physical properties of the substrate (e.g. structural support, porosity, aeration) to promote composting by increasing the void volume of the substrate without involving into the biochemical process of composting due to its inert properties. The amount of perlite used was calculated according to the initial quantity and moisture content of secondary sludge to be treated. Table 35 presents the characteristics of the perlite that was used in the 4th composting trial.

Table 35: The characteristics of perlite used in the 4th composting trial

Parameters	SiO ₂	Al ₂ O ₃	K ₂ O	Na ₂ O	CaO	FeO	MgO	Others
Perlite [%]	72	15	4	3	1	1	0.5	3.5

5.4.3. Pretreatment and preparation of the composting raw material

The secondary sludge was weighted and packed and then mixed manually with perlite using shovels and finally the mixture was ready to be fed into the bioreactor. Perlite was also weighted and then mixed with secondary sludge.

5.4.4. Feeding the in-vessel bioreactor

The feeding of the bioreactor was performed using the mixture prepared as indicated in paragraph 5.4.3 via the conveyor belt in which the feedstock was loaded manually using shovels.

5.4.5. Composting process control

Composting process control involves all the actions that had been planned and carried out with respect to the operational conditions of the in-vessel bioreactor.

5.4.5.1. Programming the plc of the in-vessel bioreactor

The operation of the agitation, hydration and aeration systems was performed by the PLC automatic control system. These operations can also be performed manually. The programming of each operation during the 4th composting trial is given below:

Programming the frequency and duration of the substrate's agitation:

For the first 8 days of the process the motor was set to operate 4 times per day (every 6 hours) for 5 minutes each time. For the next 7 days the motor was programmed to operate 3 times per day (every 8 hours) for 3 minutes each time and for the rest days of the process it operated 2 times per day (every 12 hours) for 2 minutes each time.

Programming the frequency and duration of the substrate's aeration:

For the first 8 days of the process the fan was set to operate 4 times per day (every 6 hours) for 5 minutes each time. For the next 7 days the fan was programmed to operate 3 times per day (every 8 hours) for 3 minutes each time and for the rest days of the process it operated 2 times per day (every 12 hours) for 2 minutes each time.

Programming the frequency and duration of the substrate's hydration:

The hydration system operated manually during the 4th composting trial although it could be programmed to operate automatically. The substrate was subjected to hydration whenever it was considered appropriate according to its moisture content that had been recorded from the daily measurements. The duration of the hydration

operation as well as the water flow were also adjusted according to the moisture content of the substrate that had been recorded from the daily measurements

5.4.5.2. Measuring the temperature of the substrate

Compost heat is a by-product of the microbial breakdown of organic material. Figure 35 presents the temperature evolution in the 4th composting trial as has been recorded.

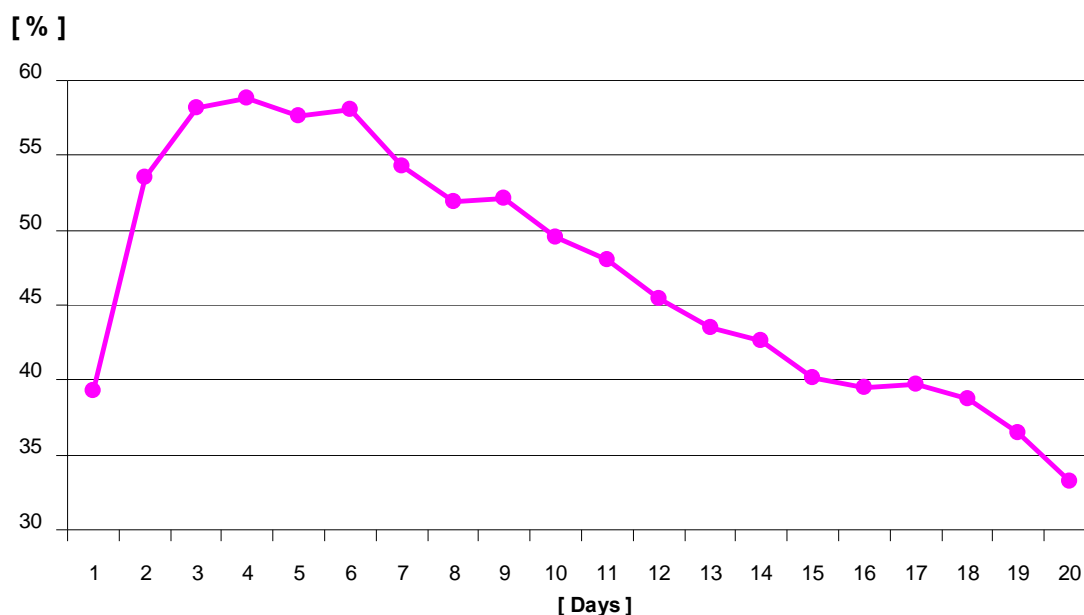


Figure 35: Temperature profile during the 4th composting trial

According to Figure 35 the substrate passed from an initial mesophylic phase (<40°C) to a thermophylic stage after the 1st day of the initiation of the composting process. Due to the initial fast decomposition of the soluble, readily degradable compounds present at the secondary sludge and due to the self-insulating qualities of the compost material the temperature of the substrate rose rapidly and reach to a maximum of 58.8°C on the 4th day. Elevated temperatures (>50°C) were maintained within the bioreactor for 8 continuous days (2nd to 9th day) which is sufficient time for the sanitization of biosolids from potential pathogenic microorganisms that exist in the composting material. Only after all substrate, capable of promoting microbial activity, had been used up did the temperature begin to fall to reach a second mesophylic phase on the 15th day of the process while the maturation process took place during this last stage of composting.

5.4.5.3. Measuring the moisture of the substrate

Optimum moisture content ranges from 40% to 60% whereas low (<30%) or high (>65%) moisture inhibits the development of composting processes. Figure 36 presents the moisture evolution in the 4th composting trial as has been recorded.

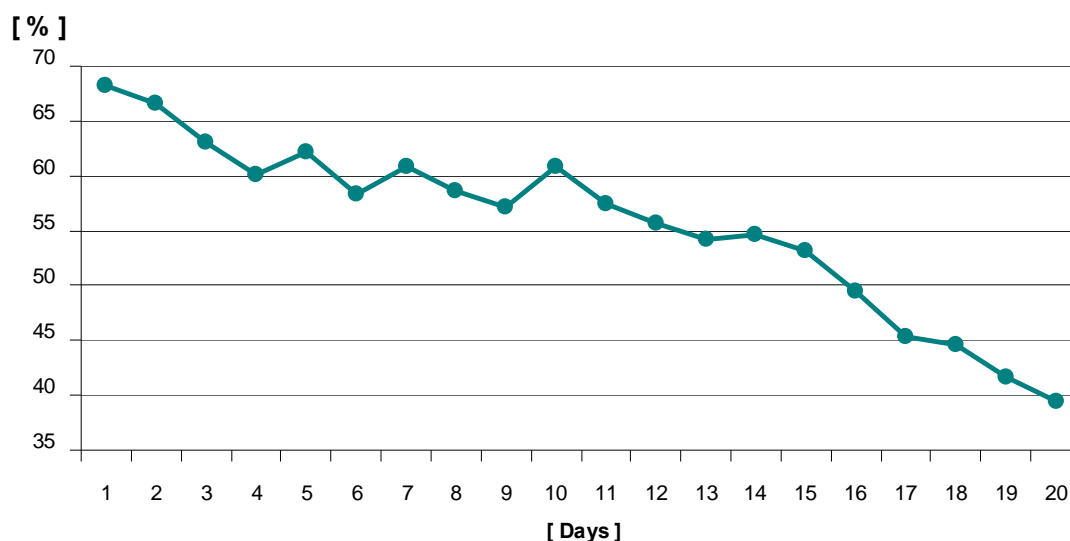


Figure 36: Moisture content profile during the 4th composting trial

According to Figure 36 the initial substrate had a moisture content of 68.2% since secondary sludge that was used as primary raw material did not undergo dewatering. The presence of readily degradable organic matter present in sludge, in conjunction to sufficient substrate's agitation and oxygen supply enhanced the metabolic activities of microbes and caused the temperature to rise rapidly leading to moisture content reduction at the initial stage of the process. The moisture content was sustained at high levels for the majority of the composting process due to the manual hydration of the substrate. The water quantity in the substrate kept decreasing throughout the duration of the trial reaching at 39.5% at the final day of the process on the 20th day.

5.4.5.4. Measuring the oxygen content of the substrate

The readily degradable components of the raw materials are metabolised during the initial period of composting. Therefore the production of heat and the need for oxygen are greatest at the early stages and then steadily decrease. Without a constant supply of oxygen, the process will slow down and if there is insufficient oxygen, the process

can become anaerobic. As has been mentioned composting process takes place even in low oxygen content (5%) however, oxygen concentrations greater than 10% are consider optimal for maintaining aerobic conditions and thus sustaining aerobic microbial activity. Figure 37 presents the moisture evolution during the 4th composting trial.

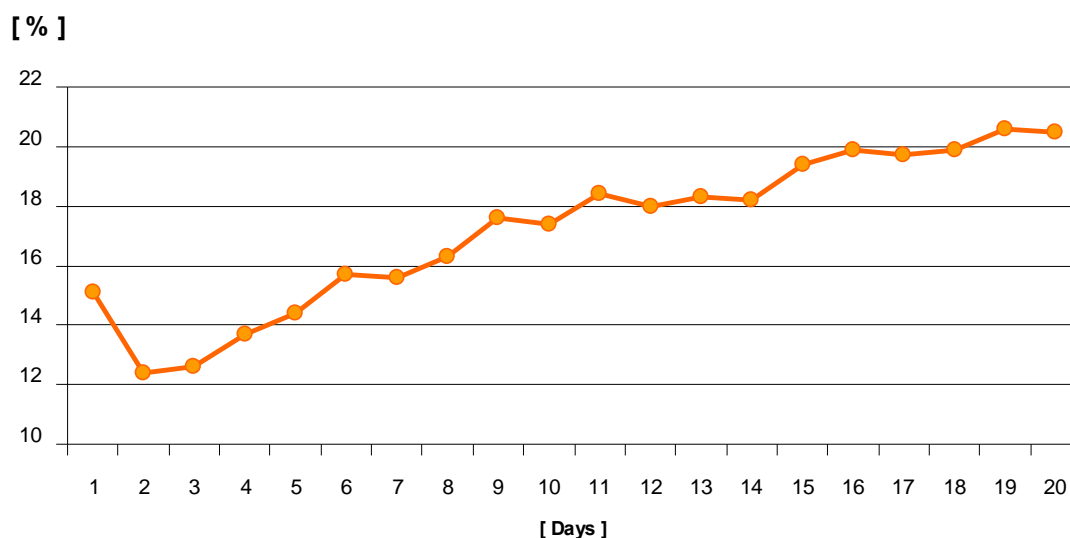


Figure 37: Oxygen profile during the 4th composting trial

According to Figure 37 the oxygen concentration of the substrate was maintained at high levels ($> 12.0\%$) throughout the duration of the 4th composting trial. The lowest oxygen concentration values were recorded during the thermophilic composting phase due to the high microbial activity of aerobic microorganisms which consume oxygen for the decomposition of the organic matter. The increased oxygen level and the incremental tendency of oxygen concentration during composting indicate that the agitation, aeration and hydration of the substrate were sufficient in promoting aerobic conditions in the bioreactor. From the 14th day onwards the substrate passed to a second mesophilic phase in which the rate of oxygen demand was reduced whereas the oxygen content was maintained above 19% until the end of the process indicative of the low rate of the organic matter decomposition.

5.4.5.5. Reprogramming the plc of the in-vessel bioreactor

The plc of the in-vessel bioreactor was not reprogrammed during the 4th composting trial.

5.4.5.6. Monitoring and maintenance during composting

Throughout the duration of the composting process the electromechanical equipment and the rest of the bioreactor's components and infrastructure were being monitored daily in order to maintain their operation status. The regular maintenance of all the electromechanical equipment of the system was performed according to the suggestions of the manufacturer (e.g. lubrication, cleaning the filter of the ventilation system, cleaning the head of the piping system, leachate removal). In addition, daily cleaning of the floor and surface of the bioreactor was performed in order to obtain high hygiene level for the personnel.

5.4.5.7. Disruption and startup the operation

No disruptions of the bioreactor's operation occurred during the 4th composting trial which lasted for 20 days.

5.4.5.8. Restoration of malfunctions and maintenance of the bioreactor

No malfunctions occurred during the 4th composting trial

5.4.6. Sampling and analysis of the substrate

Substrate samples were taken at regular time intervals (every 4 to 7 days) throughout the 4th composting trial for the examination of pH, total carbon, total nitrogen, nitrates ($\text{NO}_3^- - \text{N}$) and ammonium nitrogen ($\text{NH}_4^+ - \text{N}$).

5.4.6.1. pH

Figure 38 presents the evolution of pH values during the 4th composting trial.

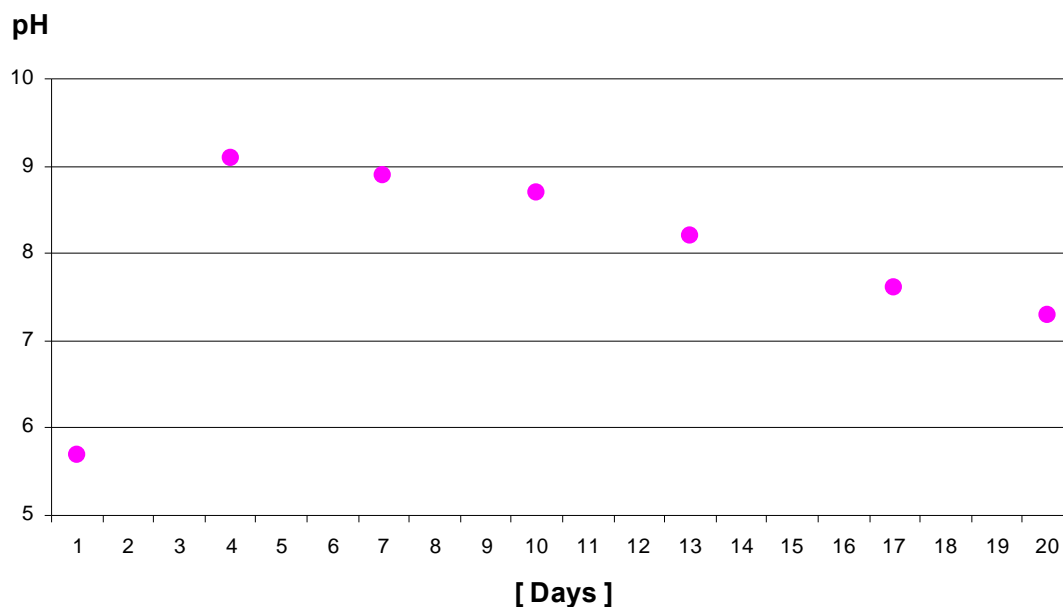


Figure 38: Changes in pH values during the 4th composting trial

The pH values increased during the initial stage of composting from 5.7 on the 1st day to 9.1 on the 4th day probably due to ammonia release through the degradation of proteins by cellular enzymes called proteases. Then through ammonia volatilization and oxidation to nitrates the ammonia content decreased and thus the pH value gradually dropped from 9.1 to 7.3 at the final day of the process.

5.4.6.2. Total Carbon

The changes occurred in the total carbon content during the 4th composting trial resemble to the changes of total carbon content during the 2nd and 3rd composting trial. Figure 39 presents the evolution of the carbon content during the 4th composting trial.

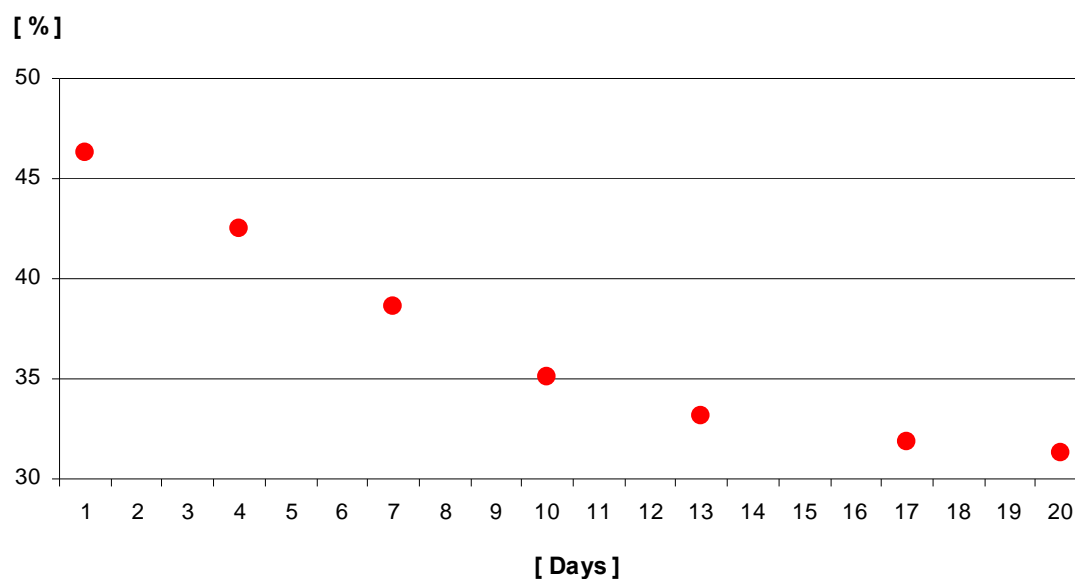


Figure 39: Changes in total carbon content [%] during the 4th composting trial

The carbon loss was attributed to the bio-oxidation of the labile organic matter originated from secondary sludge. That loss accounted around 13.18% on the 13th day since the initiation of the process. From that day onwards the rate in which carbon was being consumed decreased due to the depletion of carbon compounds capable of promoting intensive microbial activity. At the end of the process the carbon has been reduced to 31.29% achieving a 15.03% carbon loss throughout the duration of the 4th composting trial. In regard with the carbon content the end product resulting from the 4th composting trial is of good quality [19].

5.4.6.3. Total Nitrogen

Figure 40 presents the total nitrogen content evolution during the 4th composting trial.

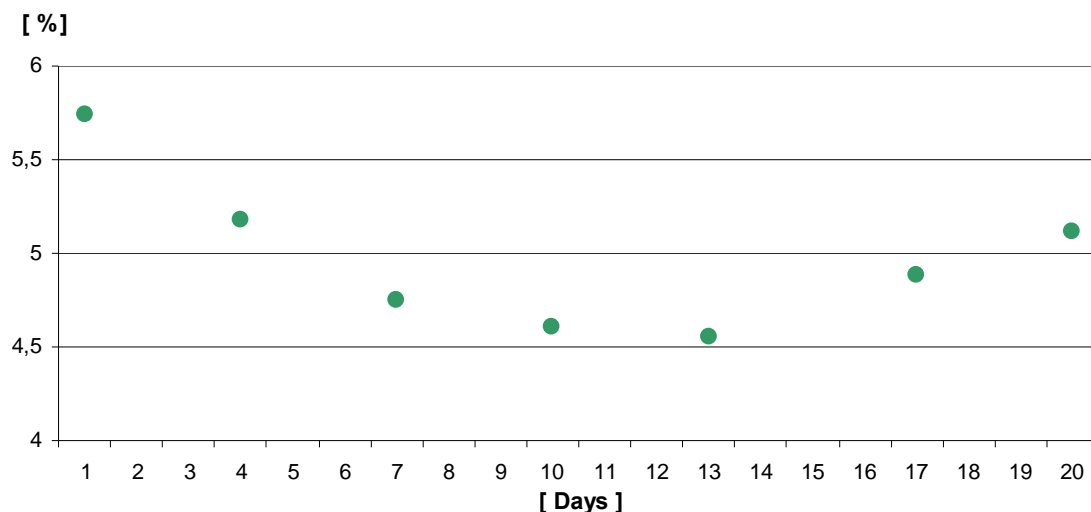


Figure 40: Changes in total nitrogen content [%] during the 4th composting trial

The 4th composting trial acquired the highest nitrogen content in comparison to the previous trials due to the origin of the composted raw material which comprised only of secondary sludge and perlite as an additive. The collected secondary sludge had increased nitrogen content as has been shown in Table 32 whereas the carbon content is low proportionally to the nitrogen content. According to Figure 40 the nitrogen content in the beginning of the process was 5.74% of which a significant amount was lost as ammonia and/or ammonium as the composting kept developing due to the excess of nitrogen in the substrate. Studies have shown that during the first phase of the process the combination of high ammonium concentration, high temperatures, and raised pH lead to high ammonia losses. Also, high aeration of the composting piles increases the rate of volatilization of the ammonia formed [65]. As has been stated the level nitrogen losses by ammonia volatilization varies and can amount up to 50% of the initial total nitrogen depending on the raw material that is used for composting [22, 23]. On the 13th day nitrogen content acquired its minimum value of 4.55%, from that day onwards and as the substrate passed from the thermophilic stage to a second mesophilic phase the nitrogen content started to increase. This increase is probably attributed to the processes of nitrogen fixation by the activity of azotobacters which recover a proportion of nitrogen that has been lost as ammonia [2, 28]. The nitrogen content at the end of the 4th composting trial on the 20th day was 5.12%.

5.4.6.4. Ammonium Nitrogen ($\text{NH}_4^+ - \text{N}$)

The ammonium evolution during the 4th composting trial followed the same pattern as the previous trials. Figure 41 presents the changes of ammonium content during the 4th composting trial.

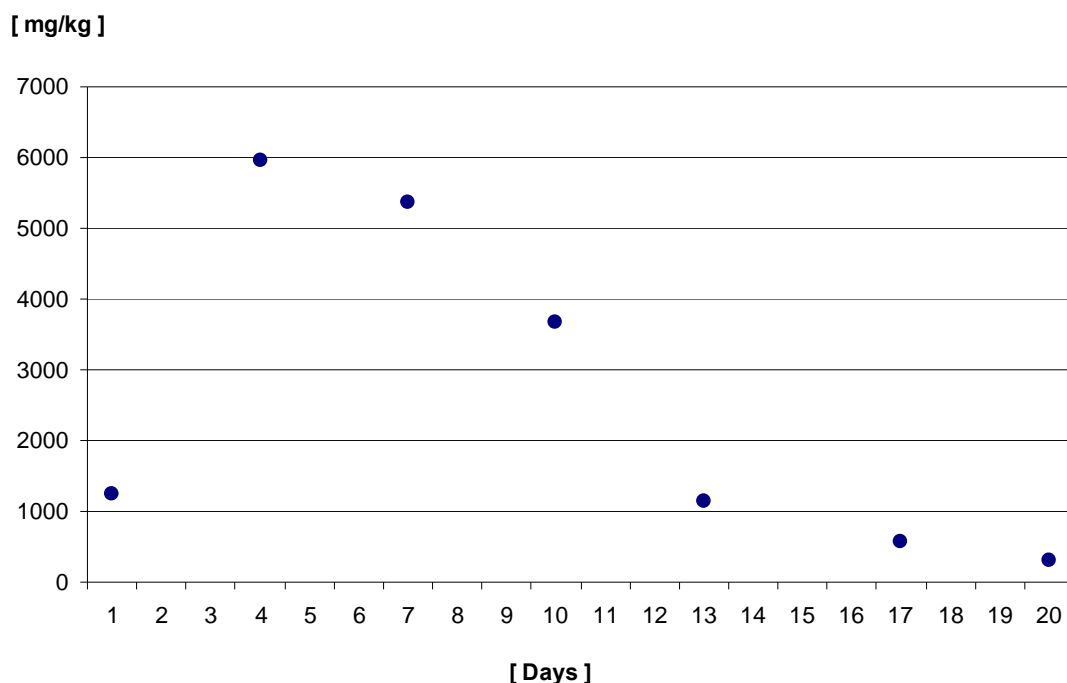


Figure 41: Changes in ammonium concentration [mg/kg] during the 4th composting trial

According to Figure 41 ammonium was formed during the initial phase of organic matter degradation reaching to a maximum of 5963.90mg/kg on the 4th day of composting. The maximum ammonium concentration in the 4th trial was the highest value among all the composting trials performed and it is attributed to the excess of nitrogen that was present to the composted material (secondary sludge). Then through ammonia volatilization and oxidation to nitrates by the action of nitrifying bacteria and by the presence of sufficient oxygen (Figure 37) the concentration of ammonium presented a steep decrease. At the end of the process on the 20th day ammonium concentration has been diminished to 304.10mg/kg. It must be noted that the presense of perlite did not assisted the ammonia selectivity as in the case of zeolite in the 3rd composting trial.

5.4.6.5. Nitrates ($\text{NO}_3^- - \text{N}$)

Besides ammonium nitrogen, nitrates concentration also changed during the 4th composting trial in the same manner as in the previous composting trials. Figure 42 presents the changes of nitrates content during the 4th composting trial.

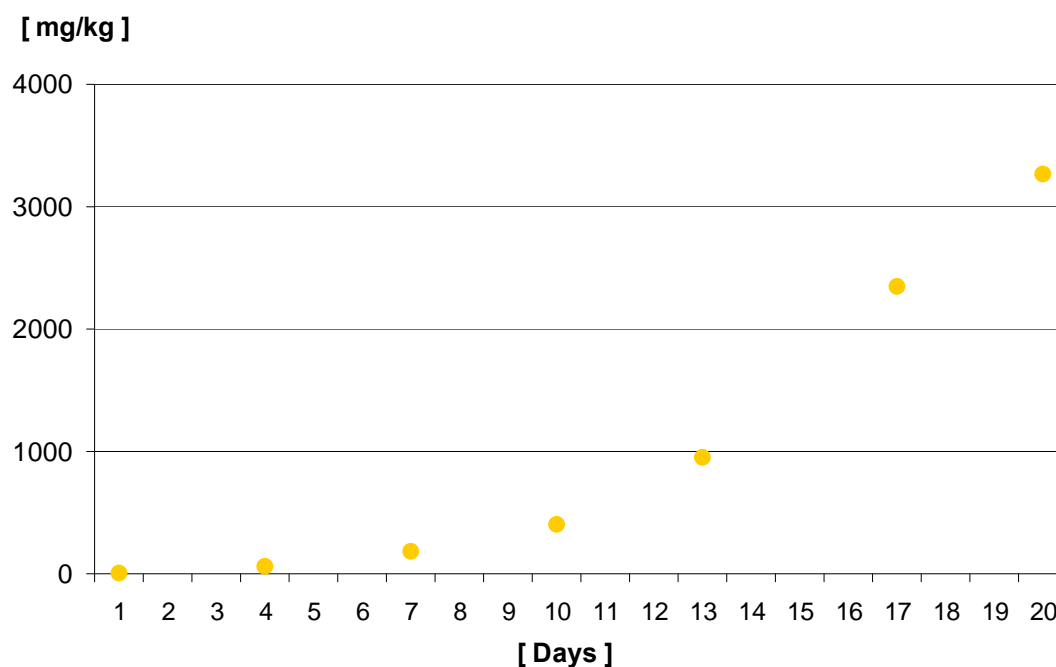


Figure 42: Changes in nitrates concentration during the 4th composting trial

Heterotrophic nitrification was present at the early phases of the process since the production of nitrates at elevated temperatures and increased ammonium concentration was almost exclusively the work of heterotrophic-nitrifiers reaching at 55.2mg/kg, 171.8mg/kg and 401.5mg/kg on the 4th, 7th and 10th day respectively [34, 67, 68]. Autotrophic nitrification was detected on a later stage of the process since this type of nitrification is markedly affected by high temperature as well as excessive quantities of ammonia which barrier the growth of autotrophic-nitrifiers (e.g. nitrobacteria). Thus on the 10th day onwards the nitrates concentration increased dramatically up to 3257.0mg/kg at the end of the process on the 20th day.

5.4.6.6. C:N Ratio

The optimal carbon to nitrogen ratios for the microbiological decomposition of organic material in composting processes is within the range of 20 to 40. Although it was expected that the substrate would initially acquire a low C:N ratio it was

considered appropriate to examine whether secondary sludge can be effectively composted using only perlite as additive¹⁵. Figure 43 presents the C:N ratio evolution during the 4th composting trial.

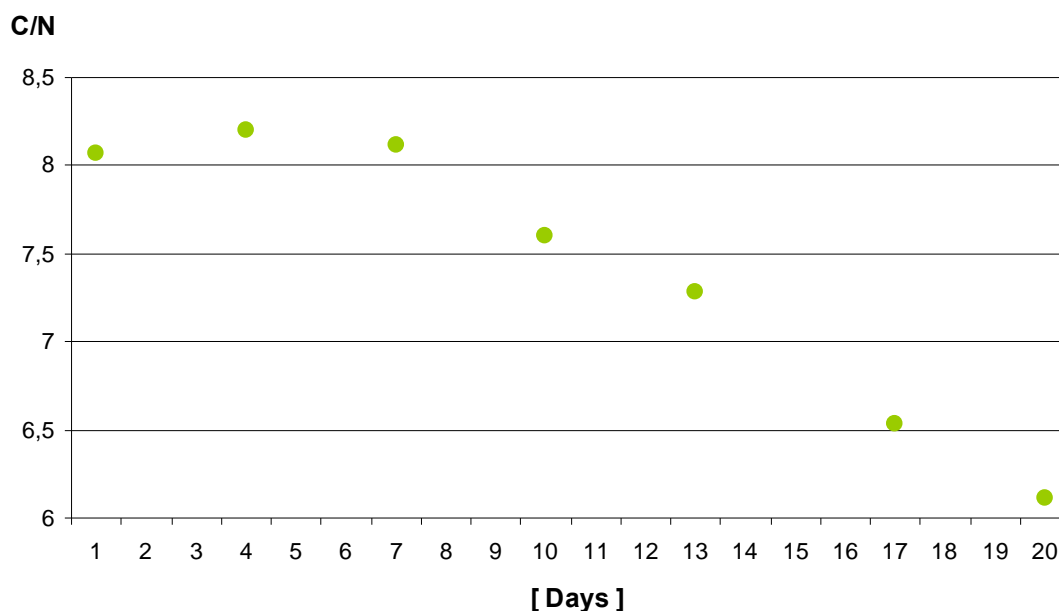


Figure 43: Changes in C:N ratio during the 4th composting trial

As has been shown the nitrogen content of the substrate was high leading to a low initial C:N ratio of 8.07. The seven first days of the process presented an increase in the C:N ratio due to increased losses of nitrogen caused by the excess of nitrogen content in conjunction to elevated temperature values. Nitrogen was lost mainly through ammonia volatilization which was trapped by the biofilter but also through the produced leachates in which a proportion of the soluble forms of ammonium escaped due to the hydration of the substrate. As the substrate passed to the second mesophilic stage and then to the maturation phase the C:N ratio decreased linearly which is attributed to the nitrogen fixation due to the activity of autotrophic bacteria, azotobacters, which recover a proportion of nitrogen. At the end of the 4th composting trial the C:N ratio had been reduced to 6.11.

5.4.7. Sampling and analysis of the substrate's leachate

Table 36 presents the evolution of the leachate parameters examined during the 4th composting trial.

¹⁵ mainly to assist aeration by giving void volume to the substrate

Table 36: Characteristics of leachates resulting from the 4th composting trial

Parameter/Time	3rd day	8th day	13th day	17th day
pH	8.0	8.9	8.7	7.8
BOD ₅ (mg/L)	72705	63949	37108	16757
NH ⁴⁺ - N (mg/L)	3802	4920	3213	708
NO ³⁻ - N (mg/L)	22.47	162.19	381.65	1473.64
Cd (mg/L)	0.00102	0.00083	0.00058	0.00051
Cr (mg/L)	0.01321	0.012853	0.00997	0.01103
Cu (mg/L)	0.24328	0.20791	0.16581	0.21438
Ni (mg/L)	0.02862	0.02258	0.01990	0.02351
Pb (mg/L)	0.05860	0.05617	0.05106	0.04671
Zn (mg/L)	1.724642	1.70854	1.91483	2.12897

During the early stages of composting the pH values were quite alkaline and reached to a maximum of 8.9 on the 8th day which was gradually decreased at 7.8 on the 17th day. The initial sample of leachates derived from the 4th composting trial acquired an increased initial BOD₅ value of 72.7g/L on the 3rd day which was reduced to 16.8g/L on the 17th day, approximately 1/4 of the initial BOD₅ value. Ammonium and nitrates in leachates followed the same pattern as the ammonium and nitrates of the substrate indicating that the composting process was well monitored since the conditions were in favor of nitrification. Ammonium ions concentration presented an increase during the first days of composting reaching to a maximum of 4920mg/L on the 8th day. From that day onwards ammonium ions concentration kept reducing and on 17th day their concentration in leachates was 708mg/L. Nitrates in leachates presented a significant increase throughout the duration of the 4th trial. On the 3rd day their concentration was limited at 22.47mg/L while on the 17th day it has been increased approximately 65 times reaching 1473.64mg/L. Table 36 shows also that heavy metals such as Cd, Cr, Cu, Ni, Pb and Zn were present in the leachates throughout the process. The metal concentrations were much below the indicative values for wastewater disposal as shown in Table 7.

According to BOD₅, ammonium and nitrates values obtained for the leachates of the 4th composting trial it can be concluded that leachates had a heavily polluted organic and inorganic load which needs to be treated before its final disposal. The quantity of leachate production during the 4th trial was limited and stored to an impermeable reservoir for sufficient period of time to be stabilized at lower values through aeration.

Although composting can be assessed for sludge treatment and reuse through land application, leachate remains an environmental problem since the treatment of solid waste through composting results in transferring the problem from solid to liquid phase. Leachate must be treated accordingly in order to be disposed to a water recipient based on the suggested standards shown in Table 7. However Directive 91/271/EEC seeks to harmonise measures relating to the treatment of wastewater at Community level in order to protect the environment from any adverse effects due to discharge of such waters

5.4.8. Removal and collection of the composting end product

The removal and collection of compost was performed on the 20th day of the composting process through the removal portals that are situated at the bottom part of the bioreactor. To facilitate the discharge of the end product the agitation system was operated for small intervals. The compost was collected manually using shovels and it was spreaded in an open area outside the building where the bioreactor is installed where it remained for five days to further reduce its moisture content (during that time the end product was being regularly agitated manually). Finally the compost was stored in order to be used to the open field experiments

5.4.9. Sampling and analysis of the produced compost

The examined physicochemical parameters of compost produced in the 4th trial are listed in Table 37.

Table 37: Physicochemical parameters of the compost produced in the 4th composting trial

Parameter	Compost
Dry Solids (% d.s.)	60.5
pH	7.3
Total Carbon (% d.s.)	31.29
Total Nitrogen (% d.s.)	5.12
C:N ratio	6.11
NO ₃ - N (mg/Kg.d.s)	3257.00

NH ₄ ⁺ - N (mg/Kg.d.s)	304.10
Total P as P ₂ O ₅ (% d.s.)	4.22515
K as K ₂ O (% d.s.)	0.53814
Ca as CaO (% d.s.)	4.71073
Mg as MgO (% d.s.)	0.93060
Cd (mg/Kg d.s.)	0.54460
Cr (mg/Kg d.s.)	13.00632
Cu (mg/Kg d.s.)	110.93610
Ni (mg/Kg d.s.)	10.05946
Pb (mg/Kg d.s.)	25.83016
Zn (mg/Kg d.s.)	730.18767

The water content of compost produced in the 4th trial was 38.8% which is within the optimum water content for composts (35% to 45%) and inhibits intensive microbial activity. In addition the moisture level is higher than 25% making compost more easily to be incorporated into the soil [2, 3, 43]. The pH was near neutral (7.30) indicating a good quality compost and within the suggested range of 6 to 8.5 [2, 3, 4, 5, 6] for the development of bacteria and fungi [69]. The total carbon was subjected to an overall 15.03% reduction throughout the 4th composting process while the remaining carbon content at the end of the process was 31.29% which is considered to be a good value [20].

With respect to the ammonium and nitrates concentration during the 1st composting trial, Figures 8 and 9 showed that when the substrate passed from the thermophilic stage to a second mesophilic, the mesophilic microorganisms that convert ammonium to nitrates flourished. The appearance of significant quantity of nitrates at the final day (886.48mg/kg) in conjunction to low ammonium concentration (302.62mg/kg) is an indicator of a maturing compost in which aerobic conditions were maintained during composting. Therefore the ratio of ammonium over nitrates to the end product can be used as indicator to assess the degree of maturity. In the case of the 1st composting trial the acquired ration was 0.24 while a ratio lower than 0.5 is indicative of mature compost [72].

As has been shown in Figure 42 nitrates concentration presented a significant increase due to the activity of autotrophic nitrobacteria which oxidize ammonium compounds into nitrates in the presence of oxygen-rich environment reaching to a maximum of 3257mg/kg at the end of the process. On the other hand Figure 41 showed that ammonium concentration was reduced significantly and by the end of the 4th composting trial it was around 304mg/kg. The evolution of nitrates and ammonium concentration showed that oxidizing factors persisted during the process while the low ammonium nitrates ratio of 0.09 is indicative of compost maturity since composts acquiring ratios lower than 0.5 are characterized as fully mature composts [72].

As has been mentioned compost acquiring high C:N ratio can cause nitrogen immobilization upon amendment to soil while too low C:N ratio can cause ammonium toxicity [44, 45]. Researchers have suggested various ideal C:N ratios from lower than 12 to lower than 25 [46, 47, 48] but the optimal value is often dependent on the initial feedstock [49]. The 6.41 ratio obtained in the 4th composting trial is the lowest ratio obtained among all trials and according to the aforementioned states it may be toxic to plants. However phytotoxicity tests that will be performed in Task 4 will show whether the increased nitrogen incorporated into the 4th compost inhibits plant growth and seed germination.

The macro-elements are used in the greatest quantities by plants especially nitrogen, phosphorous and potassium which are the nutrients most often applied through commercial fertilisers. According to Table 37 the macronutrients content of compost produced in the 4th trial were sufficiently high and in accordance to literature review (Table 9) suggesting that compost would enhance plant growth with respect to the nutrients content supply. In regard with the heavy metal concentration the finished compost complies with the EU states suggested heavy metal limits (Table 11) and it is also in accordance to the stringent German standards for maximum heavy metals concentration for intensive compost (Table 10) with the exception of Cu¹⁶ and Zn¹⁷. In addition, the 4th compost is in accordance with the environmental quality classes with respect to the heavy metal content presented in Table 13.

¹⁶ German standards Cu<50mg/kg, 4th compost Cu= 82.5mg/kg

¹⁷ German standards Zn<200mg/kg, 4th compost Zn=730.2mg/kg)

Heavy metals speciation

Furthermore, analysis of heavy metal speciation in compost was implemented (Table 38, Figures 44 and 45) in order to estimate the accumulation level of heavy metals in plants by evaluating the percentage of heavy metals that is dissolved in water and in weak organic acids (KNO₃, EDTA).

Table 38: Heavy metals speciation in compost resulted from the 4th composting trial [mg/kg]

Heavy Metals	Total	Dissolved in H ₂ O	Dissolved in KNO ₃	Dissolved in E.D.T.A	Total dissolved metals	Total dissolved [%]
Cd	0.54460	0.00267	0.00643	0.01059	0.01969	3.61
Cr	13.00632	0.01027	0.09919	0.07695	0.18641	1.43
Cu	110.93610	0.66468	1.20415	1.68449	3.55332	3.20
Ni	10.05946	0.02830	0.08872	0.11326	0.23028	2.29
Pb	25.83016	0.03971	0.15915	0.26245	0.46131	1.79
Zn	730.18767	3.70111	8.71142	13.47551	25.88804	3.55

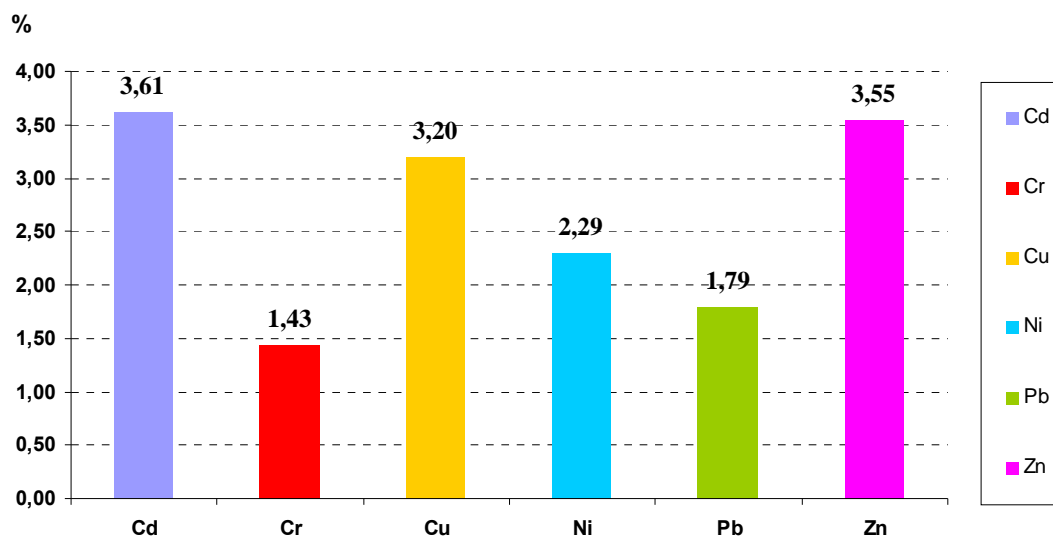


Figure 44: Percentages of total dissolved heavy metals in compost resulted from the 4th composting trial

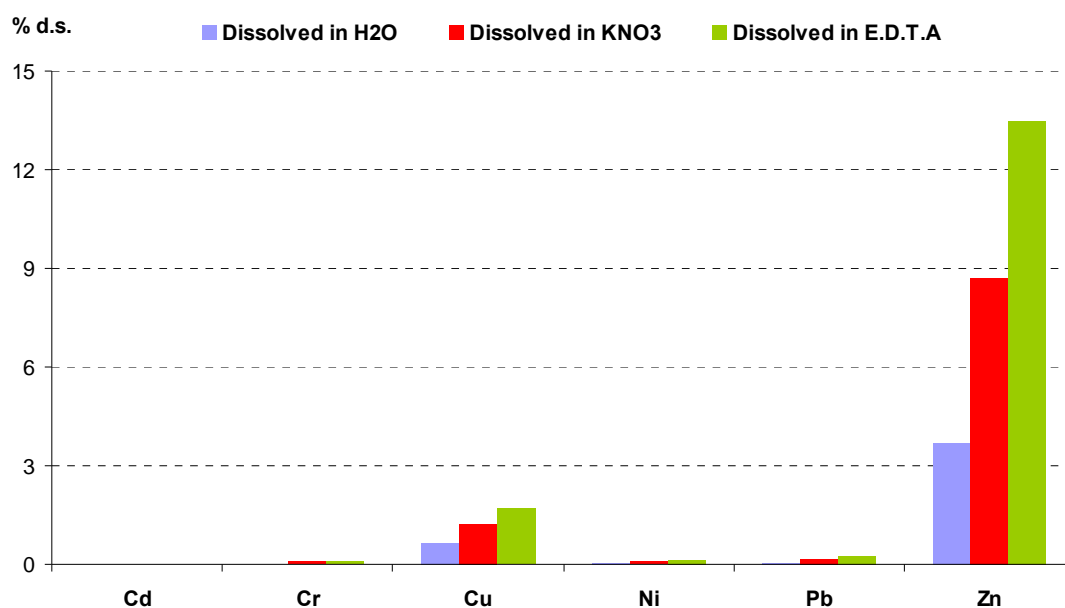


Figure 45: Heavy metals content [% d.s.] that is dissolved in water and in weak organic acids in compost resulted from the 4th composting trial

According to Table 38 and Figure 44, Cd and Zn have the highest percentages of total heavy metal dissolution, rated at 3.61 % and 3.55% respectively followed by Cu and Ni at 3.20% and 2.29% respectively but in real values the concentrations is low rating from 0.02mg/kg to 25.89mg/kg . With respect to heavy metal content that is dissolved in water, KNO₃ and EDTA, Zn and Cu are the heavy metals with the highest quantity of potential accumulation in plants as shown in Figure 45. This is in accordance to the cumulative research in Europe into the agronomic use of compost, according to which heavy metals tend to accumulate in soil and plants in the following order: Zn > Cu > Pb = Cd > Ni > Cr [59, 60].

Biological assays

To estimate the sanitization level of compost resulted from the 4th composting trial analyses have been performed to evaluate the density of total coliforms, faecal coliforms and helminth eggs prior and after the composting process (Tables 33 and 39).

Table 39: Micro-biological parameters of compost produced in the 4th composting trial

Micro-biological parameters	Compost
Total coliforms Log10 MPN/10g DS	1.60

Faecal coliforms Log10 MPN/10g DS	0.70
Helminth eggs /10g DS	<1

As has been stated there is a process-oriented ‘temperature–time’ regime in seeking to ensure a hygienic compost product. Therefore obtaining elevated temperatures (>55°C) for a sufficient period results in sanitizing the end product [61]. During the 4th composting trial temperatures higher than 55°C were obtained for approximately 5 consecutive days, as shown in Figure 35 (3rd to 7th day). The temperature values reached during the composting process appeared to be responsible for the elimination of the pathogenic microorganisms as shown in Table 39 in comparison to Table 33 [12]. Therefore it can be inferred that the 4th composting trial had been performed successfully in sanitizing biosolids such as sewage sludge since the levels of pathogenic microorganisms detected at the end of the process were significantly lower than initially [12, 13, 14].

5.4.10. Preparing the system for the implementation of the next composting trial

After the removal and collection of the compost the electromechanical components of the system were monitored and maintained while the whole system was cleaned up thoroughly. The maintenance and monitoring of the system were carefully planned and carried out according to the instructions specified in the operation and maintenance manual.

6. Concluding Remarks

An extensive presentation of the experimental work in relation to the composting process and products obtained has been given in the previous sections. In this section the results are summarized and conclusions are drawn.

Table 40 shows the composting trials (four in total) and their composition as well as the analysis of the feeding material used for each run. For the 1st trial, primary sludge was used with sugar beet leaves (the ratio being approximately 1:1). No secondary sludge was available in the area of El Jadida, while sugar beet leaves are found in excessive amounts since sugar beet along with wheat are the primary cultivations that are being practiced in Doukkala region. For the 2nd trial primary sludge, sheep manure, sugar beet leaves and straw have been used. These materials are characterized as BOW. All the above organic waste is found in abundance in the area. For the 3rd trial, primary sludge and cow manure were used as organic waste and zeolite as an additive. The role of additives is to assist the composting process without participating in the biodegradation process. They usually increase the void volume of substrate and thus better aeration and hydration is achieved. For the 4th trial, secondary sludge from a private food industry and perlite as additive was used. The above combinations are mainly based on the different waste streams that are found in abundance in the area. With respect to the chemical characteristics of the materials (Table 40) sludges acquired relatively low solid content ranging from 21.45% to 32.16% on dry basis. The high water content of sludges and the absence of dewatering installations near the area where sludges were produced was an issue of concern but with the addition of other dried materials this problem was overcome. In addition, heavy metals were found in low quantities and this was in favor of producing a good quality end product at the end of each composting trial. The temperature profiles of the composting processes developed very satisfactorily for all trials. Initially, a mesophilic temperature around 35°C developed followed by the thermophilic area 50°C to 60°C, both indicating that the processes were being well developed. The moisture content initially varied significantly from trial to trial but during the process the moisture was controlled in order to be kept within the required values 50% to 60%. The moisture content falls to lower values towards the end of the composting process. For a successful composting process oxygen plays a very important role for

the degradation and stabilization of the organic substrate under aerobic conditions. All trials were very successfully controlled by maintaining the oxygen content at high levels ensuring that adequate oxygen was being diffused within the mass of the substrate. The values were much above the 5% oxygen content which is considered as a minimum value to avoid anaerobic environment. The successful control of aeration, hydration and temperature were achieved by the control system that constitutes an essential component of the bioreactor.

During the composting process there are many physical, biological and chemical changes of the substrate and leachate is formed and removed from the bioreactor. In all trials the leachate produced contained high quantities of biodegradable organic matter expressed as BOD₅. The BOD₅ concentration varied from 72,705mg/L to 50,984mg/L at the initial stage of composting and then a gradual decrease was observed leading to BOD₅ values at the end of the process that ranged from 8,958mg/L to 16,757mg/L. Also ammonium and nitrates concentrations were very high much higher than the limits set for wastewater disposal. Heavy metals are also found in the leachate but much higher quantities are associated with the substrate as it can be seen from the metal content of the feeding material and the compost product. Leaching quantities were limited and they were very much dependent on the moisture content within the bioreactor. In the present study leachates were collected and stored in order to be disposed off in an appropriate way. It must be emphasized that during composting especially in full scale application there will be a need for a leachate treatment plant.

The pH value in all composting trials showed variation starting from near neutral moving to alkaline values during the full development of composting and then back neutral values indicating that the composting process was towards the end. Also the C:N ratio significantly decreased during the development of the composting process. The initial C:N values for the 1st, 2nd, 3rd and 4th trial follow the order 16.74, 19.61, 13.30 and 8.07 indicating that different BOW can give different C:N ratios. As the composting process was developing the C:N ratios were decreasing and obtained values below 12 indicative of good quality compost, the values being 10.08, 11.87, 9.79 and 6.11 respectively. It is worth mentioning that the four trials have exhibited good performance as far as composting retention time is concerned following the

cycles/stages that are expected for composting processes e.g. mesophilic (1st stage), thermophilic, mesophilic (2nd stage), normal conditions. From the results it is shown that the main composting process is nearly over within 15 to 18 days with the exception of the 1st trial. The rest of the time is mainly for the completion of the composting and could be performed outside the bioreactor and thus optimizing its use. The 4th trial, which included the treatment of secondary sludge and additives, followed the shortest cycle but acquired the lowest C:N values.

The most important part of the work is to achieve a good quality compost for the different trials and the different BOWs and additives used. The C:N ratios obtained reflect to a good quality compost that acquire ratios lower than 12 which is considered as a very good value for finished composts. The relatively high content of nutrients (NPK) for all trials indicate that the compost produced can be used for agricultural purposes while the ammonium to nitrates ratios ranging from 0.09 to 0.24 are within the suggested limits of fully mature composts. One of the limiting factors of compost application in land is their heavy metals concentration. In this project extensive work has been performed to quantify the metal concentration of the feedstock as well as the metal concentration in leachates, substrate and finished compost. The results indicate that the metal concentration is quite low. It should be noted that the heavy metal concentration does not really reflect the readily available metal concentration. For this purpose metal speciation studies were performed. Only low percentage 1.4% to 6.2% of the various metals is available in dissolved forms. The presence of available metals is very low when zeolite was used indicating that the additive has trapped the mobile forms of the metals. The density of pathogenic microorganisms in the produced compost was insignificant due to the fact that the elevated temperature values obtained during composting resulted in the elimination of the pathogens.

Table 40: The physicochemical characteristics and the quantity of the raw material used in each composting trial

	1st Trial		2nd Trial				3rd Trial			4th Trial
Parameter	Primary sludge [700kg]	Sugar beet leaves [650kg]	Primary sludge [520kg]	Sheep Manure [250kg]	Sugar beet leaves [490kg]	Straw [50kg]	Primary sludge [700kg]	Cow Manure [180kg]	Sugar beet leaves [450kg]	Secondary sludge [1500kg]
Dry Solids (%)	27.8	51.65	32.16	84.37	53.74	90.27	31.3	92.56	52.10	21.45
pH	6.8	7.1	6.3	8.5	7.2	7.1	6.5	9.2	7.1	5.7
Total C (% d.s)	10.5	59.94	11.03	47.71	58.60	59.08	12.6	45.43	61.50	46.32
Total N (% d.s)	1.76	3.52	1.91	2.83	3.43	0.94	1.65	2.65	3.48	5.74
C/N ratio	5.97	17.03	5.77	16.86	17.08	62.85	7.64	17.14	17.67	8.07
Total P as P ₂ O ₅ (% d.s)	1.300	0.240	1.3942	0.9310	0.2470	0.1721	1.0715	0.5620	0.2605	3.68
K as K ₂ O (% d.s)	0.691	3.524	0.9670	4.2683	3.4673	1.9890	0.8540	3.7545	3.5878	0.46
Ca as CaO (% of d.s)	4.873	3.387	4.5235	6.8071	3.4418	1.1122	4.2507	6.9176	3.6205	4.13
Mg as MgO (% of d.s)	2.339	1.381	2.2073	1.7388	1.5807	0.5666	2.0860	1.2601	1.5463	0.82
Cd (mg/Kg d.s)	0.986	0.113	0.8218	0.5937	0.1590	0.1305	1.1731	0.7858	0.1022	0.4820
Cr (mg/Kg d.s)	18.410	0.828	25.2105	10.4570	0.5719	0.6012	21.6286	13.8386	0.8056	12.9371
Cu (mg/Kg d.s)	141.471	7.924	135.2713	17.3205	12.2520	6.6361	175.3108	24.65726	13.414	98.3961
Ni (mg/Kg d.s)	24.305	0.895	26.9106	11.1481	0.9365	1.0037	31.4635	16.0901	1.3201	9.7721
Pb (mg/Kg d.s)	81.094	0.504	63.3828	2.8133	0.3043	0.3260	91.6719	2.17552	0.3830	21.9202
Zn (mg/Kg d.s)	237.894	48.916	251.8231	34.8637	31.9314	23.909	225.5545	45.4673	36.5834	593.7208

Table 41: Physicochemical characteristics of composts produced from each composting trial

Parameter	Compost 1	Compost 2	Compost 3	Compost 4
Dry Solids (% d.s)	63.9	63.8	61.2	60.5
pH	7.31	7.28	7.03	7.3
Total Carbon (%)	28.84	33.84	23.80	31.29
Total Nitrogen (%)	2.86	2.85	2.43	5.12
C/N Ratio	10.08	11.87	9.79	6.11
NO ₃ ⁻ - N (mg/Kg d.w)	1286.48	998.43	388.12	3257.00
NH ₄ ⁺ - N (mg/Kg d.w)	302.62	207.73	38.65	304.10
Total P as P ₂ O ₅ (% d.s)	0.7584	0.9264	0.79322	4.22515
K as K ₂ O (% d.s)	3.0118	3.9505	3.21070	0.53814
Ca as CaO (% d.s)	4.7107	5.4785	5.74819	4.71073
Mg as MgO (% d.s)	2.1266	2.0931	2.02320	0.93060
Cd (mg/Kg d.s)	0.4739	0.59735	0.72949	0.54460
Cr (mg/Kg d.s)	8.1537	11.19308	13.51757	13.00632
Cu (mg/Kg d.s)	64.2264	54.17476	82.47363	110.93610
Ni (mg/Kg d.s)	10.8509	11.96504	17.38393	10.05946
Pb (mg/Kg d.s)	34.0357	20.03207	38.15530	25.83016
Zn (mg/Kg d.s)	132.8445	109.24231	118.22976	730.18767

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