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Compost Maturity Evaluation and its Significance to Agriculture

¹Chukwujindu M.A. Iwegbue, ²A.C Egun, ³F.N. Emuh and ⁴N.O. Isirimah

¹Department of Chemistry, ²Agric Education Unit,
Delta State University, Abraka, Nigeria

³Department of Crop/Soil Science, Delta State University, Anwai Asaba

⁴Institute of Pollution Studies, Rivers State University of Science and Technology,
Port Harcourt, Nigeria

Abstract: One of the main obstacles to the successful utilization of compost in agriculture is the lack of reliable quality criteria and understanding the transformation of organic matter, which take place throughout the whole process. Proper evaluation of composts maturity essential for establishment of such criteria. Because of the diversity of origin of composts. It is impossible to use a single method to evaluate compost maturity. It is best done by a combination of methods. A good index of compost maturity should have an overall trend describable by a monotonic function. Methods for evaluating composts maturity can be categorized into (1) chemical analysis (C/N, CEC and nitrification) (2) physical analysis (odour and temperature) (3) spectroscopic analysis (FTIR and ¹³C-NMR) (4) plant bioassay (germination and plant growth) (5) chromatographic analysis (sephadex fractionation) (6) degree of humification (HI, DH and HR) (7) microbial analysis (respiration). These tests vary in precision, simplicity, costs, duration and approach. Plant bioassays are considered the most direct test for compost maturity since it shows the effect of compost maturity on plant growth and other maturity tests are correlated with plant growth performance. Spectroscopic methods, sephadex fractionation and degree of humification give an insight to the feasibility of starting material to be transformed, the level of humification and the quality of the humic fraction and the efficiency of a given composting process.

Key words: Maturity index, degree of humification, humic acid evolution index, phytotoxicity

INTRODUCTION

Land application of compost from municipal waste could be one of the most economical and attractive methods of solving two problems; waste disposal and the necessity to increase the organic matter content of soil (Giusquani *et al.*, 1988). Compost maturity is an important factor affecting the successful application of composts for agricultural purpose (Inbar *et al.*, 1990; Mathur *et al.*, 1993; Wu and Ma, 2002). The improvement of commercial viability of composts is still a pressing problem with most compost plants. One of these is the lack of routine standard for rapidly evaluating the product quality and plant performance. The result is that the control of attained degrees of stabilization of composts are often too empirical and unreliable (De Nobili and Petrusi, 1988). At the same time, up till now the problem of finding a suitable test for assessing compost maturity in the marketed products have not been solved.

A good index for the evaluation of compost maturity should have an overall trend describable by a monotonic function; otherwise its measurement would not give

unequivocal results. It is also necessary that stabilization of the index is not reached too early i.e. before the end of thermophilic period (De Nobili and Petrusi, 1988). The degree of compost maturity at the conclusion of the process is critical to the marketability of the product (Butler *et al.*, 2001).

This study critically examines the various methods of measuring compost maturity in an attempt to define the most profitable and reliable methods of evaluating compost maturity.

COMPOST MATURITY, PROCESS AND PROBLEMS ASSOCIATED WITH USE OF IMMATURE COMPOSTS

Composting evolved through an active and maturation phase. The active phase of the process is characterized by intense microbial activity, which assure the stability of organic matter avoiding product deficiencies i.e., phytotoxicity due to the presence of easily decomposable compounds, thereafter the microbial population shift to those that decompose cellulose and

hemicellulose primarily actinomycetes and fungi. The compounds are harder to decompose and yield less energy. Thus, the rate of decomposition is reduced and less heat is given off. This is usually considered curing (Henry and Harrison, 1996). Maturation, which starts at end of thermophilic phase, converts the already digested organic matter into humic compounds (Adami *et al.*, 1997; Chen and Inbar, 1993).

Compost stability and maturity are comprehensive properties including indicating the degree of organic matter decomposition and potential phytotoxicity caused by insufficient composting. Currently, there is no official definition for the two terms (Wu and Ma, 2002).

The term "compost maturity" can be generally referred to degree of completion of the composting process. The technical term "compost maturity and compost stability" are not synonymous (Iannotti *et al.*, 1993). Maturity refers to the degree of humification of the material. Compost stability refers to the level of activity of microbial biomass (Butler *et al.*, 2001). Since composting is a microbial process, compost stability and maturity are the result of microbial activities. It is important to note that the chemical composition and organic matter decomposition status of the compost will play an important role in determining the microbial status (Wu and Ma, 2002). Kurihara (1984) defined compost maturity as condition in which the composted material can be stored without creating a nuisance or can be applied without causing problems.

The maturity of compost is obscure (Sughara and Inoko, 1981) and stabilization occurs within indefinite period of time (De Nobili *et al.*, 1986). Application of immature composts can induce anaerobic conditions due to continued decomposition of the immature compost (Harada and Inoko 1980a; Mathur *et al.*, 1993). The microbial biomass utilizes oxygen in the soil pores to break down the materials. This inturn can deprive plant roots of oxygen and lead to the generation of hydrogen sulphide (H_2S) and nitrite (NO_2^-) (Mathur *et al.*, 1993). Another problem associated with the use of immature compost to soil is the prevalence of high (25:1 or greater) carbon to nitrogen ratio in such materials (Iglesias Jimenez and Prez Garcia, 1989). Immature composts with high C to N ratio can induce nitrogen saturation in plants as microbes scavenge soil N to make up the deficit (Butler *et al.*, 2001). The other problems include phytotoxicity due to the presence of organic acids such as the intermediate products of decomposition. Acetic acid and phenolic compound, in particular, may suppress seed germination, inhibit root growth or suppress crop yield (Sughara and Inoko, 1981; Zucconi *et al.*, 1981a; b; Chanyasak *et al.*, 1983a, b; Butler *et al.*, 2001).

METHODS OF EVALUATING COMPOST MATURITY/STABILITY

Because of the diversity in the origin of the compost, it is impossible to use a single method to evaluate the maturity of given compost. It is best to use a combination of methods (He *et al.*, 1992). Many tests for compost maturity are relative tests i.e. the feedstock or the intermediate composted material is compared with a final product. The tests differ in simplicity, duration and approach. These test methods could be classified as follows (i) chemical methods (ii) biological methods (iii) microbiological methods (iv) spectroscopic methods (v) degree of humification (vi) chromatographic and (viii) physical methods (He *et al.*, 1992; Henry and Harrison 1996).

Chemical parameters

Cation exchange capacity: Cation Exchange Capacity has been heavily investigated as potential indicator for compost maturity. The functional groups responsible for the CEC are mainly the carboxyl and phenolic groups. The contribution of the carboxyl group is about 55% and the phenolic and enolic group account for another 35%. The cation exchange capacity in organic material increases as function of humification due to the formation of carboxylic and phenolic functional groups (Lax *et al.*, 1986). Carboxyl groups are formed by the oxidation of lateral chain of the aromatic rings or hydrolysis of ester or lactones (Lax *et al.*, 1986).

Harada and Inoko (1980 a) estimated the degree of maturation of city refuse compost by measuring the Cation Exchange Capacity (CEC). The CEC of city refuse compost increased for the first 7 days after the commencement of the fermentation process, decreased for the next 2 days and then increased gradually up to the end of the piling period. A high significant correlation was noted between CEC and C/N ratio of the city refuse ($r = -0.903$). Regression analysis yield the relationship. In $CEC = 7.02 - 1.02 \ln C/N$. The authors considered municipal refuse with a CEC greater than or approximately 60 (C mol kg^{-1}) of the ash free basis to be sufficiently matured for application to cropland.

Cation exchange capacity was determined by saturating 25 different composts samples with acid resin and removing H^+ ions (Estrada *et al.*, 1987). Values were assayed with or without other chemical parameters of organic fraction, resulting in a useful index of maturity. The CEC value range from 19.7 to 66.7 C mol kg^{-1} . The organic fraction had the highest CEC value (150-150 C mol kg^{-1}) and generally underwent significant change with time, compared with the mineral fraction (10 C mol kg^{-1}) that did not change much during

composting. The ratio of CEC to total organic matter (CEC:TOM) values correlated with the index of maturity obtained from other chemical parameters; degree of decomposition, humic acid and fulvic acid (extraction and polymerisation rate) and colourimetric index of the humic extract (More and Sana, 1987). Jacas *et al.* (1987) reported a high correlation ($r = 0.66-0.94$) between compost age and CEC, despite different feedstock. The authors recommended that CEC test as routine maturity test for its precision, simplicity, low cost and speed. Harada *et al.* (1981) found that cation exchange capacity of city refuse increased up to 70-80 C mol kg⁻¹ during 5 to 8 weeks and thereafter showed a constant value. It was confirmed therefore, that the C/N ratio, the total nitrogen content, the ratio of the carbon in reducing a sugar to the total carbon and the CEC considerably reflects the changes in organic constituents during the piling process and represented useful parameters for estimating the degree of maturity. Saharinen (1998) reported that CEC should be calculated per unit ash since this prevents the overestimation of the number of cation exchange sites on composted material.

Harada and Inoko (1980 b) developed a method of measurement of cation exchange capacity of compost. This method is similar in principle to those developed for the measurement of CEC of plant roots. This method involves washing 200 mg of milled compost with HCl to replace the exchangeable cations. After the excess HCl has removed washing with water, Ba(OAc)₂ solution (pH 7.0) is added to the sample and the mixture was left to stand overnight. After filtration, the compost was leached with another Ba(OAc)₂ solution and then the released proton is titrated with standard NaOH solution using a potentiometer. This method given reproducible and accurate value but requires a particular apparatus. It takes a relatively long time to carry out the whole procedure. A simplified method, in which the sample is left to stand for only 1 hour for the equilibration with Ba(OAc)₂ solution and the titration is carried out using thymol blue as indicator has been recommended.

C/N ratio: Carbon to Nitrogen ratio has been used as indication of the potential of compost maturity. Jimenez and Garaia (1992) has compared initial to final C/N ratios in an attempt to relate them to maturity. The C/N ratio of an MSW compost was 30:1 for raw material and decreased to 13:1 with composting (Charpentier and Vasout, 1985).

Inoko *et al.* (1979) analysed the organic composition of municipal refuse composts produced in Japan from the standpoint of their suitability for application to land. The results indicated that the C/N ratio ranged from 19 to 31. There is much evidence that composted materials having a C-N ratio less than 20 cause no N starvation when

applied to soil. They recorded decreases in total C, hemicellulose and cellulose and increase in total N, crude ash and lignins during maturation of city refuse compost. The authors reported that hot water-soluble organic matter, hemicelluloses, cellulose and reducing sugar also varied widely with feed-stock and from plant to plant, immature samples with a high C-N ratio had higher content of cellulose and hemicellulose determined as reducing sugar.

Total C, C/N ratio, the content of cellulose and hemicelluloses and the ratio of C in reducing sugar total C decreased during composting of municipal solid waste (Harada *et al.*, 1981). Le Bozec and Resse (1987) concluded that MSW composts reached maturity at a C/N below 20:1. The authors assigned appropriate composting time frame in accordance with method and season. Twenty weeks in summer and 22 weeks in winter for windrow, 17 weeks in summer and 15 weeks in winter for static aerated piles; 12 weeks in summer and 8 weeks in winter for aerated pile with periodic turning. Sughara and Inoko (1981) suggested that C/N ratio is not suitable for assessment of compost maturity when inoculants are added.

Chanyasak and Kubuta (1981) focused their attention of the water-soluble phase of the compost, where microorganisms are actually active. They found that the ratio between organic and organic carbon nitrogen in the water extracts of composts was very variable during the first phase of maturation, but decreased to uniform final value of 4.5 to 6.5 for well matured composts, regardless of the type of starting raw material. Unfortunately this ratio is low (5 to 6) even in raw sewage sludge and this parameter cannot be used to assess a standard measure of maturity of these materials.

Garcia *et al.* (1991) suggested that the organic matter content of the water extract, the water soluble carbon/water soluble nitrogen ratio, the sugar content and biodegradability index can be used as indices of maturity since, in addition to the decrease of the values with time, similar values were obtained in the four cases studied at the end of the composting and maturation process. The water soluble carbon/organic nitrogen ratio in the extracts can also be considered as a good index of compost maturity since this ratio reached similar values (5) at the end of the processes in all composts. Chefetz *et al.* (1996) reported that the change in the C/N ratio and ash content reflects organic matter decomposition and stabilisation during composting. The C/N ratio decreased rapidly from an initial value of 28 in the raw materials to 18 after only 20 days. The ratio continued to decrease, albeit less sharply to 12.2 after 60 days, from this point on the C/N ratio stabilized at a value of about 12 (11.8-12.8) for the remainder of the process.

Nitrification: Nitrification is the oxidation of NH_4^+ to NO_2 and NO_2 to NO_3^- by microorganism. Nitrification occurs when the competition for N is less (Henry and Harrison, 1996). Chefetz *et al.* (1998) reported that the total amount of soluble N decreases during composting. Decreased levels of organic N represent mineralization. During the thermophilic stage, ammonium-nitrogen level of composts were high, indicating a high rate of degradation of organic N compound by microorganisms. The high ammonia levels during thermophilic decomposition can produce an odour problem. During Naturation, the ammonium N levels continue to decrease, while the nitrate levels increases. The ratio of $\text{N-NO}_3^-/\text{N-NH}_4^+$ increased to 4 at this stage. The increasing as an indicator of compost maturity. Finstein and Miller (1985) defined compost maturity by nitrification, the authors considered that a compost is matured and ready for use when NO_2^- and/or NO_3^- appears. Riffaldi *et al.* (1986) considered decreasing then stabilizing NH_4^+ and increasing then stabilizing NO_3^- suitable for evaluation of compost maturity.

pH: In the earliest stage of composting pH value decreases due the production of organic acids derived from the intense fermentation carbohydrates. Afterwards, the pH began to rise and reached 9.02 at the thermophilic period. This increase results from the release of ammonia due to the start of the proteolytic process. (De Nobili and Petrusi, 1988). Chefetz *et al.* (1998) reported that the pH during exhibited at typical pattern for composting process. It drop temporarily during thermophilic stage due to accumulation of organic acids reflects the high rate of OM degradation and these acids are used later on as substrate by other microorganisms. During the cooling down and maturation stages the pH drop to a neutral value. pH cannot be considered a good parameter to assess compost maturity as its overall trend is not describable by a monotonic function (De Nobili and Petrusi, 1988).

DEGREE OF HUMIFICATION OF ORGANIC MATTER

Humification process is the sum of the degradation of lignin to aromatic units, which occurs mainly during the thermophilic phase and the subsequent biosynthesis from the condensation and polymerization of aromatic units and cellular debris (Tomati *et al.*, 1996). The humification mainly consists of the polymerization and rearrangement of sub/units of with molecular weights, mediated by microbial populations growing in the composting waste (Tomati *et al.*, 2001).

Compost properties strongly depend on the level of stabilization. Rolletto *et al.* (1985) suggested the adoption of a set of chemical parameters to characterized organic matter. They include, in addition to percentage of carbon extractable in pyrophosphate (defined as humification ratio), the humic/fulvic acid ratio, nominal molecular weight distribution of extracts and a "humification index" define as the percentage of humic carbon on total carbon. It seems reasonable to infer that such chemical parameters are useful in monitoring compost maturation. The authors, for instance, suggested the use of minimum value of 3.50% referred the humification index, which shows, however, a greater variability among five different composts, ranging from 4.31 to 18.57%.

Today, the most reliable texts for evaluating compost maturity are those base on the separation of non-humified (NH) from humified fraction (HA, humic acids and FA, fulvic acid) (Sequi *et al.*, 1986; De Nobili and Petrusi, 1988; Saviozzi *et al.*, 1988). The first parameter proposed was humification index HI i.e. the ratio between the nonhumified NH and humified fraction (HA = Humic Acid; FA = Fulvic Acid).

$$\text{HI} = \frac{\text{NH}}{\text{HA} + \text{FA}} \quad (1)$$

HI reaches values less than 0.5 for humified substrate (Sequi *et al.*, 1986).

Two other parameters for evaluating the levels of humification of compost have been proposed (Ciavatta *et al.*, 1988). These are (i) the degree of humification DH and (ii) humification rate (HR) given by the equations.

$$\text{DH}\% = \frac{\text{HA} + \text{FA}}{\text{TEC}} \times 100 \quad (2)$$

and

$$\text{HR}\% = \frac{\text{HA} + \text{FA}}{\text{TOC}} \times 100 \quad (3)$$

Where TEC = Total extractable carbon and TOC = Total organic carbon.

Both HR and DH% tend to increase as humification proceeds. All these parameters indicate the stabilization reached by the end product (Tomati *et al.*, 1996).

De Nobili *et al.* (1986) focussed on the ratio between non humified and humified fraction in pyrophosphate extracts during maturation of sewage sludge compost. The ratio is higher than 1 in the raw material but decreases to a value less than 1 with time. It is assumed that this reflects quantitatively the degree of stabilization of organic matter in the sludge. Analytical

isoelectrofocussing (IEF) experiments show that humified fraction of the raw sludge focalize only in the pH gradient regions lower than 5.0, while in older sludge samples, there is a progressive strengthening of band in the region between pH 6.5 and 5.0. This is at least partially similar to IEF patterns of soil organic matter. IEF patterns are suggested to reflect stabilization of organic matter in sewage sludge from a quantitative point of view.

De Nobili and Petrusi (1988) monitored the stabilization of a pile of urban waste over on period of five months. The humification index HI of the organic matter extracted by 0.1 M $\text{Na}_4\text{P}_2\text{O}_7$ plus 0.1 M NaOH was found to decrease linearly with time during the therophilic phase from about 1.4 to 0.4. Turning of the pile caused a drop in the HI value, but the index increased again each time the temperature of the static pile reaches 65°C. The values of the humification index in water extracts (HI_w) reach a maximum after 4 days in the superficial layer and 11 days at the depth of 30 cm. HI_w values are found to decrease hyperbolically with time during the thermophilic period.

The humification of olive mill waste compost was assayed. The degree of humification, the humification rate and the humification index which respectively reached the values of 78, 37.8 and 0.28% after 2 months (Tomati *et al.*, 1996).

Adani (1995) developed a New Organic Matter Evolution Index (OMEI) (formally known as organic matter stability index) by determining the humic acid content in samples of composts, both as they stand and when subject to treatment with polar and non polar solvents and acid hydrolysis in order to remove the psauo humic substance that interfere with the analytical determination. The index, which registered values between zero and one, easily relate to the degree of stability and maturity of organic matter.

The method has been found very useful for direct measurement of the degree of stability ($\text{SI} < 0.6$) and maturity ($\text{SI} > 0.8$) of organic matter, as well as the effectiveness of analytical method proposed for determining the humified fraction of organic matter. Maturity of organic matter in respect of SI value greater than 0.8 describe the attainment of a state in which the humification processes are extremely advanced, this represent maximum stage of organic matter evolution in aerobic condition (Soltner, 1988; Adani, 1995). Compost at this stage can be utilized in any situation without exhibiting phytotoxicity (Chen and Inbar, 1993; Adani, 1995).

The significance of OMEI as related to the composting process degree was tested in a full-scale windrow process for 239 days. Analytical data from many samples collected during the process and kinetics were in

agreement regarding the sufficient degree of stability and maturity reached by the compost obtained. The OMEI at the end of the process was low (OMEI = 0.46). This confirmed its validity for composting degree evaluation. (Adani *et al.*, 1997).

CHROMATOGRAPHIC METHODS

Composting is a process that evolves towards the biosynthesis of acid. Therefore the molecular weight of humic fraction lead us to estimate the feasibility of the starting materials to be transformed, the level of humification, the quantity of humic fraction and the efficiency of given composting process (Tomati *et al.*, 2000).

The decrease in low molecular weight fractions and transformation into fractions with high molecular weight has been reported by Chanyasak *et al.* (1982). Rolletto *et al.* (1985) Daz Burgos and Polo (1991) and Garcia *et al.* (1992). These authors observed that a molecular weight of about 5-10 KDa disappeared while fractions with higher molecular weight increased over composting time. The greatest fraction had a molecular weight of about 50 KDa because of the Gel filtration columns used (G-25, G-50, G-75, G-100) did not permit the separation of fraction with a larger molecular weight (Tomati *et al.*, 2000). Gel chromatograms of water extracts from composting pulp and paper sludge with straw showed an initial increase of molecular weight followed by a decrease and disappearance (Saviozzi *et al.*, 1987). The same trend occurred with C, N., volatile acid and amino acid content. As composting proceeds, HA molecular weights increased by polymerization of subunits with lower molecular weight.

The elution profiles of obtained by gel selphadex (G150) showed the disappearance of fraction with molecular weights < 50 KDa and contemporary increase of fraction with molecular weight > 10² KDa. In this range, two fractions can be separated, first one (A₁) with molecular weight in the range of 10² KDa to 210² KDa and second one (A₂) with molecular weight > 10² KDa. During composting the ratio A₂/A₁ tend to reach a constant value, which indicates the evolution towards the polymerization of HA. The ratio A₂/A₁ is known as humic acid evolution index (HAEI). This indicates the transformation of polymers having molecular weights in the range 10²-210² KDa into polymers having molecular weight greater than 210² KDa. It varies with the material composted, the composting process and represent the maximum possible degree of humic acid polymerization (Tomati *et al.*, 2000). This can be assumed as a more reliable index for evaluatating compost maturity.

SPECTROSCOPIC METHODS

A more complete picture is obtained by considering the chemical and physical properties of humus like compound produced. Non destructive methods, in particular ^{13}C -CP-MAS-NMR (Carbon 13 Cross Polarization Magic Angle Spinning Nuclear Magnetic Resonance) spectroscopy and Fourier Transform infrared Spectroscopy (FTIR) have been widely applied to gather information on the nature of humic acids, their evolution and changes in functional groups (Schnitzer and Preston, 1986; Inbar *et al.*, 1990; Chefetz *et al.*, 1996).

The FTIR spectra represent several stages during composting. The main absorbance bands were: a broad bands at 3300 3400 cm^{-1} (H-bonds, OH groups), two distinct peaks at 2930 and 2850 cm^{-1} (C-H asymmetric, C-H stretch of $-\text{CH}$), a slight shoulder around 1716 cm^{-1} (C=O of COOH), a wide peak at 1650 cm^{-1} (C=C in aromatic structure, COO^- , H-bonded C=O.) and small peak at 1540 cm^{-1} (Amide II bonds,) a tpeak at 1518 cm^{-1} (C=C of aromatic rings), a sharp peak at 1450 cm^{-1} (C-H deformation of CH_2 or CH_3 groups), a small peak at 1419 cm^{-1} (CH_2 , COO^-), a small peak at 1375 cm^{-1} (CH_3 , COO^-), a pronounced peak at 1222 cm^{-1} (aromatic C, C-O stretch) and two peaks at 1126 and 1031 cm^{-1} (C-O stretch of polysacharride) (Baes and Boom, 1989; Inbar *et al.*, 1990; Stevenson, 1994; Chefetz *et al.*, 1996, 1998).

The distinct change in the spectra resulting composting was a reduction of 1560 cm^{-1} peak which probably due to relatively rapid biodegradation of the amino chain. The aromatic region and the 1800 cm^{-1} peaks became sharpen during composting. Peaks in the aliphatic region at 2930 and 2850 cm^{-1} decreased, while the 1450 cm^{-1} peak increased (Chefetz *et al.*, 1996).

The changes in the IR spectra is monitored by calculating the ratio of the between intensity of major peaks (Inbar *et al.*, 1990; Chefetz *et al.*, 1996). The peaks at 2930 cm^{-1} , 2850 cm^{-1} , 1650 cm^{-1} , 1560 cm^{-1} and 1050 cm^{-1} were usually considered. The ratio 1650/2930 (Aromatic C/Aliphatic C) increased from 0.88 to 1.10, the ratio 1650/2850 (Aromatic C/Aliphatic C) increased from 0.79 to 1.54, the 1650/1050 ratio aromatic C/polysaccharide) increased from 2.39 to 2.80 and the 1650/1560 ratio (aromatic C/amide II bond) increased from 0.94 to 1.5. The changes reflect a decrease in level of polysaccharide, aliphatic and amide components and an increase in levels of aromatic structure in mature compost. Increase in aromatic structure is relative and it result from decomposition of aliphatic moieties (Chefetz *et al.*, 1996).

The linear correlation between the aromatic to aliphatic peak ratio 1650/2930 and the C/N ratio exhibited

$R^2 = 0.936$ calculated from $y = 1.263 - 0.014x$. This correlation indicates the FTIR is a useful and reliable index for compost maturity evaluation and analysis of OM during composting.

CP-MAS ^{13}C -NMR is the only physical method holding some promise for the reliable characterization of organic matter and providing useful information on transformation of organic matter during the composting (Chen and Inbar, 1993).

The major peak exhibited in the CP-MAS ^{13}C -NMR spectra were at 30 ppm. (Methylene C), 55 ppm (methoxyl C), 72 ppm (carbohydrate or aliphatic alcohols), 105 ppm (anomeric carbons of polysaccharides), 130 pm (C-substituted aromatic C) 150 ppm (O-substituted aromatic and 170 ppm (carboxyl C) (Kogel-Knabner *et al.*, 1991; Chefetz *et al.*, 1996).

The distinct changes in the spectral resulting from decomposition of organic matter was reported by Chefetz *et al.* (1996). These changes were; reduction of the 30 ppm peak; appearance of the 55 ppm peak as shoulder in the mature compost and an increase of the aromatic region peak (130 and 150 ppm peaks). The authors also noted the following changes (i) total aliphatic C (0-112 ppm) decreased by 14.4% from 78.4% in the raw material to 64.0% in the matured compost; polysaccharide (60-112 ppm) decreased by 17.0% from 42.3 to 35.1% in the matured compost and alkyl groups (0-50 ppm) decreased by the 11.0% during the composting process; (ii) aromatic components, including aromatic and phenolic carbons, increased by 54 from 15.9% in the raw material up to 24.5% of the total C in the mature compost; total aromaticity increased by the 58.2 from 17.5% in the raw material to 27.7% in the mature compost; (iii) carboxyl C increased by only 9.5% and (iv) the C=O carbonyl carbon by 73.7 from 1.9% in the raw material to 33% in the matured compost.

BIOLOGICAL INDICATORS

Plant bioassay: Plant bioassays are perhaps one of the most appropriate tests for maturity because they indicate when composts may be used without phytotoxic effect. Phytotoxic response in plants has been exploited as an index of compost maturity.

Wong (1985) found that the seed germination of flowering Chinese cabbage (*B. parachinesis* Bailey) in MSW compost samples was lower than that in the controls. High growth suppression was recorded in the fresh and 2 weeks old compost; 3 weeks old compost produced 80% germination. Zero to 4 weeks old composts produced much shorter root length compared with the control. More so, the inhibitory effect shown in the fresh, 2 weeks and 3 weeks old becoming less in the 6

week old composts. The root lengths in 8 weeks compost and above were similar to that of control.

The author noted that phytotoxicity decreased with age while metal concentrations remain essentially unchanged. This suggest that metal are not the main cause of toxic response in the bioassays even though certain metals at low concentration may slow and hinder root growth. The decline in toxicity with age is due to degradable organic compounds. The inhibition of germination and root growth is associated to ethylene oxide and ammonia content.

Radish (*Raphanus sativus* L.) and ryegrass (*Lolium perenne* L.) growth bioassays revealed that immature compost samples inhibited growth. Growth of ryegrass in pot mix prepared with cured compost not amended with fertilizer was enhanced as compared to a peat control.

Garden cress (*Lepidium sativum* L.) seed germination used as indicator of phtotoxicity, revealed inhibition of germination at all compost maturity. The phytotoxicity is salt related (Iannotti *et al.*, 1994). Mathur *et al.* (1986) verified the maturity of fish waste compost using germination test for phytotoxicity, C/N and cation exchange capacity. The germination index and disappearance of the high molecular component after 20 (days) were suggested as useful parameter for assessing compost maturity because equilibrium reached (Saviozzi *et al.*, 1987; Henry and Harrison, 1996). A germination index higher than 80-85% indicated the absence of phytotoxicity in 2 months old compost (Riffaldi *et al.*, 1986). A highly sensitive germination index can be obtained by multiplying germination and root growth, in that low toxicity (i.e. at concentrations as low as 25 ppb of compounds associated with decomposing organic matter) affects root growth; high toxicity inhibit germination. This method produced concentration dependent responses and differentiated among stages of maturity (Zucconi *et al.*, 1981a).

A bioassay based on the growth of cucumber (*Cucumis setivus* L. v Dilla) plant revealed that plant grown in media containing fresh (14 days) compost exhibited inhibited growth compared to plant grown on older compost. The dry weight of plants grown samples taken after 28 to 84 day of composting statistically similar. The dry weight of plant grown in 112 and 132 days compost were significantly higher than all the others. The highest plant dry weight was achieved after 112 days of composting. This correspond to the sample in which maximum amount of humic acid were present and to the time at which the 1650/1560 drift infrared peak ratio has stabilized at the maximum. The linear correlation between the 1650/1560 ratio and plant dry weight exhibited a

$R^2 = 0.81$ ($Y = 0.81 + 0.548x$) and linear correlation between HA content in OM and plant dry weight exhibited a $R^2 = 0.70$ ($Y = 3.57 + 7.78x$) suggests that these parameters could indicated compost maturity (Chefetz *et al.*, 1996).

Wu *et al.* (2000) reported that compost exhibited significant decrease of phytotoxicity with curing on tomatoes seed. The authors reported 90-97% germination rate for Wislow sample which correspond well with low CO₂ evolution whereas, Sunset samples yielded low germination rate (31-55%). The indicated that Sunset-samples were still phototoxic to tomatoes seed despite the low evolution of carbon dioxide. The germination rates in Sunset sample are associated with the feedstock and actual composting stage. This demonstrated that stability based on CO₂ evolution and phytotoxicity based on seed germination tests are different properties (stability/maturity) and low respiration rates does not necessarily reflect low phytotoxicity.

Phytotoxicity of composts is usually determined by a seed germination index assay or by plant growth test. The main deficiency of these tests is the length of time required for their performance (Helfrich *et al.*, 1998).

Helfrich *et al.* (1998) developed a new biosensor technique in phytotoxicity studies based on oxygen consumption and fluorescence measurements of isolated freshly suspended or lyophilized chloroplast thylakaloids (*Vicia faba* L.). The new systems proved to be suitable for evaluating the function of the photosynthetic electron transport (PET) between photosystem II and photosystem I. When phytotoxic substances inhibit PET, fluorescence increased whereas oxygen consumption decreased. Germination index data as well as plant growth bioassays data exhibited high correlation with data obtained from the novel biosensor technique. The application of these methodologies based on freshly isolated or lyophilized chloroplast membranes, as rapid and sensitive test for compost phytotoxicity.

The genotoxic effect of compost was carried using the micro-nuclei test in *Vicia faba* on both aqueous or organic (5% Dimethyl Sulfoxide DMSO) extracts.

The evaluation of the genetotoxic effect of the extracts was carried out by studying the frequency of micro-nucleated cell (MC) in root meristems of *Vicia faba*; the result made evident a similar trend in the effect of induction by both aqueous and organic extracts. No genotoxic damaged was recorded for agroindustrial residue i.e., composts from olive mill pomace and tomatoes industry.

A significant increase in MC frequency was observed when *Vicia faba* seedlings were treated with extracts from the organic fraction of solid urban waste composts. The composts exhibited capacity of inducing micro-nucleated

cells. This effect is due to toxic compounds in the starting material. However, as the composting process proceeds, this toxic effect decreased to about 10 times the genotoxic potential of the starting materials (De Simone *et al.*, 2000., Tomati *et al.*, 2002). As phytotoxicity is the general term of reference to evaluate noxious effect on plants, genotoxicity could stand a good index to evaluate compost maturity.

PHYSICAL INDICATIONS

Physical sign of stability, such as loss of self-heating and general appearance; uniformity, dark colour and earthy smell, are most reliable when compared with other parameters.

Odor: Although there has not been quantitative measurements for smell (for obvious reasons of subjectiveness), it is seen reasonable that compost maintains an earthy smell while storage will be mature enough not to cause detrimental effect on plants.

Conversely, compost with an obnoxious odour suggests instability (Henry and Harrison, 1996).

Temperature: Temperature trend or reheating under laboratory conditions have been proposed as an index of maturity (Harada *et al.*, 1981; Henry and Harrison, 1996). Three typical phases of composting were observed by temperature measurements during composting (Harada *et al.*, 1981; Jimenez and Garcia, 1992; Inbar *et al.*, 1993, Chefetz *et al.*, 1996). These are (i) a mesophilic phase, during first 2 days in which temperature rose to 45°C; (ii) a thermophilic phase lasting 42 weeks in which the temperature increased to a maximum of 72%; further temperature increase was avoided by turning and mixing the compost once in week (iii) after 60 days of composting the temperature equalled that of the ambient (Chefetz *et al.*, 1996) – At this point the compost is assumed to be reasonably stable. Temperature was dismissed as dependable indication of maturity in that temperature increase may not occur if conditions are not conducive to composting (i.e., moisture limitation) (Henry and Harrison, 1996).

Colour change: Sughara and Inoko (1981) investigated colour change during composting process. The colour of the composting material changes to dark or greyish black with advancing maturity. The change in colour of materials during composting were measured using the CIE 1931 standard colorimetric system. The authors found that (i) the shape of the relative spectra reflectance curve

of rice and barley straw different from each other immediately after the straw has been dried, but gradually become smooth and similar during composting (ii) the stimulus value Y_1 which generally indicates the degree of lightness of materials, decreased rapidly during the first 42 days of piling of rice straw treated without addition of calcium cyanamide and then reached a constant value (iii) the stimulus value Y reached a constant value rather rapidly when rice and barley were treated with calcium cyanamide and (iv) the chromaticity coordinate (X_1Y), which specify Munsell notation hue and chroma hardly changed.

A positive correlation between the stimulus value Y and the C/N (with correlation coefficient 0.931 ($p < 0.001$)) was observed in rice straw treated without the addition of calcium cyanamide. Regression analysis yielded the equation $Y = 0.549 C/N + 7$ and standard error of regression was estimated to be 3.1 ($n = 9$). The authors concluded that constant values of Y observed after piling indicates the threshold of maturity of straw compost, although full maturity was seldom reached.

Watanabe and Kurihara (1982) devised a measurement method consisting of extracting humus like substance from the sample with 0.1 M $Na_4P_2O_7$ solutions and determining the colour of the extracted solution colourimetrically using a standard solution of purified humic acid. The absorbance is measured at 420 nm. The extraction rate of the humus like substance is linearly correlated with C/N ratio and materialization or immobilization (or both) of N. When municipal refuse compost has an extraction rate of humus like substance greater than 5%, it is assumed to be sufficiently matured.

The extract from fresh cattle manure compost was dark brown in colour and, as the composting proceeds, the intensity of this colour decrease to light brown. These colour change were monitored as absorbance at 465 nm. A similar curve also was obtained when soluble organic carbon was measured. As C compound that are highly available to microbes (e.g. soluble, sugar, hemicellulose, amino acids and proteins) were degraded during the thermophilic stages of decomposition, they were transformed into either CO_2 , or insoluble stable organic matter, resulting in a reduction in soluble C. The colour of water extract has often been used as a rapid indicator for dissolved organic carbon (DOC) concentration. A highly significant correlation between the water-soluble organic carbon extracted from the compost and absorbance at 465nm was observed providing a simple tool for the determination of dissolved organic matter (Inbar *et al.*, 1993).

MICROBIAL ACTIVITY INDICATIONS

As organic materials are decomposed aerobically by microbes, CO₂ is given off. Thus, the rate of CO₂ evolution has been considered as a useful measure of composting activity. Conversely as the rates significantly reduce, may be approaching maturity, if other environmental factors are limiting (N availability, moisture) (Henry and Harrison, 1996). Peaks and falls in CO₂ evolution during composting coincided with those in microbial activity (Hardy and Sivasthamparam, 1989; Henry and Harrison, 1996). Respiration bioassay used to determine stability (CO₂ and CO₂ respirometry) were sensitive to process control problem at the composting plant and indicated increasing stability with time. Spearman rank order correlations demonstrated that O₂ respirometry, water-soluble organic C and the water extract organic C to organic N ratio, significantly correlated with compost age and best indicated an acceptable level of stability. Oxygen respirometry also best predicted the potential for ryegrass growth and an acceptable level of compost maturity (Iannotti *et al.*, 1994).

The effect of compost age and sample storage was reported by Butler *et al.* (2001). The author reported that the Dewar flask self-heating test was the most useful indicator of compost maturity. This test showed changes throughout the 57 days composting period while oxygen respirometry was not sensitive to change after 29 days to composting, suggesting that this is not by itself an adequate indicator of stability. Although C.E.C. was found to increase substantially for stored sample, no clear trend seen for samples that were tested without storage. Storage primarily affected samples that are more than 15 days old.

Biological Oxygen Demand (BOD) was determined as both an indicator of compost maturity and for studying the process (Stahlschmidt, 1978). A simplified version of the Warburg method of respiration was applied with sludge compost. Measurements were recorded for the change in partial pressure of O₂ in a sealed container filled with compost. A decrease in BOD of 75% occurred within 30 days of aerobic curing. Compared with organic matter, C/N ratio, chatomin test and a growth test, it was concluded that given optimal composting conditions, maturity is reached when the BOD is < 50 mg O₂ kg⁻¹ at 50% h⁻¹ and remains so for some days. Reliability of this method depend on control of temperature and homogeneity of samples. Correlation with bioassays was suggested for purpose of quality control during the process (Henry and Harrison, 1996).

CONCLUSION

Biodegradable organic matter (OM) is the main fraction of municipal solid waste (MSW). It may represent 34 to 50% w/w of the total waste stream depending on the culture under consideration, type of waste collection used and climate etc. Recycling of biodegradable organic waste fraction can decrease the total amount of land filled and benefit of waste management in general. Composting is considered a desirable treatment option for the biodegradable fraction as it has the capacity to reduce its volume and weight by approximately 50%. Furthermore, resulting recyclable product can be beneficial to agriculture. One of the main obstacles to the successful utilization of compost in agriculture is the lack of reliable quality criteria and understanding of the transformation of organic matter, which take place through out the process. Proper evaluation of compost maturity is essential for establishment of such criteria. Because of the diversity in the origin of composts, it is impossible to use a single method to evaluate the maturity of a given compost. It is best done by a combination of methods. Many of these tests are relative tests, i.e. the feedstock or the intermediate composted material is compared with the final product.

A good index for evaluating compost maturity should have an overall trend describable by a monotonic function; otherwise its measurement would not give an unequivocal result. Tests for evaluating compost maturity differs in their simplicity, duration and approach. These test methods are categorized into (i) chemical analysis (ii) physical (iii) microbiological analysis (iv) plant bioassay (v) and spectroscopic methods and (vi) degree of humification. The most direct approach includes smelling and plant bioassay (Plant growth or germination). Phytotoxic in immature compost is due to the presence of salts, short chain volatile, primarily acetic acids. Plant bioassays are perhaps one of the most appropriate tests for compost maturity because they indicate when compost could be used without phytotoxic effects. The main deficiency of these tests is the length of time required for their performance. The new biosensors technique developed by Helfrich *et al.* (1998) is more rapid and sensitive. This new technique appears too promising.

CEC, C/N ratio and nitrification have also been proposed as methods of evaluating compost maturity. CEC and C/N ratio requires relatively short period of time. However these tests do not give information of the nature of the decomposing organic matter.

Compost properties depend strongly on the level of humification. The most reliable are those based on the separation of non-humified from humified fraction (HA, humic acid and FA Fulvic acid). These are HI, DH% and HR. HI and DH% tend to increase as humification proceeds. All these parameters indicate high stabilization reached by the end product.

Composting is a process that evolves towards the biosynthesis of humic acid. Therefore molecular weight of the humic fraction gives us an insight to the feasibility of starting product to be transformed, the level of humification, the quality of humic fraction and efficiency of a given composting process. An index that evaluates compost stability and quality through evolution of molecular weight is known as Humic acid evolution index. It varies with material composted, the composting process and represents a maximum possible degree of HA polymerization. This method is promising. A more complete picture is obtained by considering the chemical and physical properties of the humus-like compound produced. ¹³C CP-MAS NMR (Cross Polarization Magic Angle Spinning) spectroscopy and fourier transformed infrared spectroscopy (FTIR) have been useful diagnostic tools to gather information about the nature of humic acid, their evolution and changes in functional groups. The NMR and FTIR revealed an increase in aromaticity and carboxylic group and a decrease in polysaccharide and aliphaticity as the decomposition process. These findings were verified by crude fibre analysis, changes in cation exchange capacity, humic content (which increased) and a decrease in C/N during compost.

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