

Guide Standard: Treated Faecal Sludge Compost for Non-food Applications

IAPMO – I TFSC – 01: 2022



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FOREWORD

Circular sanitation is emerging as one of the key pillars of achieving the Sustainable Development Goal 6 (SDG 6) clean water and sanitation for all. Treatment and reuse of faecal sludge is beneficial in a variety of ways, including but not limited to:

- lowering risk of water-borne infections and environmental pollution
- reducing emissions and mitigating climate impacts
- providing valuable resources to improve food security, agricultural security, and (deteriorating) soil health
- contributing to economic growth through innovative circular economy-based business models

Over 100 million new toilets built under Swachh Bharat Mission (SBM) 1.0 and hundreds of new sewage treatment plants (STPs) and faecal sludge treatment plants (FSTPs) being commissioned in tier 1 and tier 2 cities are considered to be significant sources of treated faecal sludge in commercially-relevant quantities needed in a circular sanitation setup.

Market Potential for Treatment of Biosolids in India

About 1.2 billion Indians generate about 1.75 million tonnes of faecal matter every day. A KPMG and National Faecal Sludge and Septage Management (NFSSM) Alliance report (June 2020) states that more than 60% households in India are dependent on On-Site Sanitation (OSS). According to the Ministry of Housing and Urban Affairs (MoHUA), only 20% of urban local bodies (ULBs) safely manage their septage or sewage. A KPMG report states that there are about 1200 STPs at various stages across the nation. A NITI Aayog and NFSSM Alliance report (January 2021) states that over 20 states and union territories have committed to 700+ faecal sludge treatment plants (FSTPs). The treatment plants at current capacities grossly fall short of the overall treatment capacity needed.

While states are building treatment capacity to meet demand, so far the focus has been on treating black water and grey water, with little attention towards treatment of faecal sludge. Treated biosolids have potential to produce compost, briquettes, and biogas, among other materials of value. Briquettes and biogas are used as inputs in thermal processes and their direct use is considered safe. Compost for agricultural use, prepared by either drying faecal sludge or co-composting with organic waste, however, needs to be checked for safety.

The SBM Urban 2.0 guidelines state that ULBs can explore revenue streams by selling compost prepared from faecal waste. Biosolids need to be treated for pathogens, helminths, heavy metals, and other unsafe substances, before being considered safe to use for agricultural purposes and being able to generate a continuous revenue stream.

The main constraints in commercial application of faecal sludge compost are

- product description and definition, and
- widely accepted standards backed by relevant testing, evaluation, and certification schemes

The Toilet Board Coalition (TBC) strongly believes in the potential of a circular economy for sanitation, with relevant standards and certification being key to unlocking this potential. The International Association of Plumbing and Mechanical Officials (IAPMO), a global testing, standards, and certification body, with capabilities in the fields of water and sanitation, has for decades pioneered new standards that have helped safety and sustainability causes. The TBC and the IAPMO India have joined hands to develop testing and evaluation methods and guide standard for treated faecal sludge compost for agricultural applications, with or without supplementation with other forms of organic solid waste (e.g., kitchen waste).

The IAPMO India will leverage its global 'industry' standard development framework, set up a working plan, formulate a committee comprising an industry member nominated by TBC, a subject expert from IAPMO, and at least two external experts to deliver a jointly authored comprehensive document for compost from treated faecal sludge that describes a guide standard, testing and evaluation methods, and certification schemes for the agreed product class (namely, TFSC).

DISCLAIMER

The Guide Standard: Treated Faecal Sludge Compost for Non-food Applications (IAPMO – I TFSC – 01: 2022) is a set of guidelines recommended for those involved in evaluating faecal sludge compost products (having faecal sludge as primary source alone or 70% in combination of animal dung and/or kitchen waste). The provisions of this guide standard are not mandatory.

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TERMS AND DEFINITIONS

The terms and definitions described in this document pertain only to this standard.

Faecal sludge

Faecal sludge is defined as what accumulates in onsite sanitation technologies and specifically is not transported through a sewer. It is composed of human excreta, but can also have traces of other materials from an onsite containment technology, such as flushwater, cleansing materials and menstrual hygiene products, grey water from bathing, cleaning, and kitchen use, and solid waste.

Soil conditioner

A soil conditioner is defined as a product that is added to soil to improve the soil's physical qualities, usually its fertility and sometimes its mechanics.

Soil nutrient

A soil nutrient is defined as a product with minimum units of NPK and organic content that is added to soil to improve the soil's physical qualities as well as deliver nutritional benefits.

Composting

Composting is defined as a process of controlled microbial decomposition of organic matter, typically in aerobic conditions, resulting in the production of stable humus-like product.

Contaminant

A contaminant is defined as an undesirable physical, chemical, or microbiological substance or parameter in water that may have adverse effects on health or aesthetics, or both.

Non-food application

Non-food applications (in the context of TFSC) refers to applications for those plants that are not used for direct consumption by humans, such as cotton, rubber, jute, oilseeds, horticulture crops.

Total organic C

Organic matter is the most valuable component of compost for soil health improvement. Total organic carbon and organic matter are expressed as a percentage of compost dry weight. Organic carbon (C) represents about half of the organic matter weight.

Bulk density

Bulk density is dependent on soil organic matter, soil texture, the density of soil mineral (sand, silt, and clay) and their packing arrangement. It is the weight of the soil in the given volume. A compact soil has a higher value while an organic soil has a lower value. It also affects water holding capacity of the soil.

Faecal Coliforms

Faecal coliforms should be <1000/g dry weight. Faecal coliforms can survive in both aerobic and anaerobic conditions and is common in all initial compost piles. Most human pathogens occur from faecal matter and all faecal matter is loaded in faecal coliforms. Therefore, faecal coliforms are used as an indicator to determine if the chosen method for pathogen reduction (heat for compost) has met the requirements of sufficient temperature, time and mixing. If faecal coliforms are reduced to below 1000 per gram dry weight it is assumed all other pathogens are eliminated. Potential problems are that faecal coliform can regrow during the curing phase or during shipping. This is because the conditions are now more favourable for growth than during the composting process.

1. Tests shall be carried out as per methods prescribed in Annex B.
2. Unless otherwise specified, quality reagents, chemicals, and distilled water shall be employed in tests

1. SCOPE

This standard covers general safety and quality specifications and performance requirements of treated faecal sludge compost (organic supplement) for non-food applications.

2. REQUIREMENTS

- 2.1 Treated faecal sludge compost shall be a dried solid.
- 2.2 Treated faecal sludge compost shall be used as either soil nutrient (Grade A) or soil conditioner (Grade B).
- 2.3 Treated faecal sludge compost shall meet quality and efficacy parameters specified as in Table 1. The values shall be determined by methods as in Annex B.
- 2.4 Treated faecal sludge compost shall meet the safety parameters (and shall have no contamination with heavy metals and pathogenic organisms with adverse impacts on soil, plant, and human health) specified as in Table The values shall be determined by methods as given in Annex B.
- 2.5 Treated faecal sludge compost shall have a minimum of 3 months expiry period from the date of manufacture.

3. SAMPLING

The method of drawing representative samples of treated faecal sludge compost and the criteria for conformity shall be as given in Annex A.

4. TESTING

Tests for use as soil nutrient (Grade A) or soil conditioner (Grade B) shall be carried out as per testing specifications as given in Table 1.

5. PACKING, MARKING, AND STORAGE

5.1 Packing

Treated faecal sludge compost shall be packed in packaging material of low-density polyethylene or polypropylene bags or insuitable high-density polyethylene or polypropylene containers or any other packaging materials suitable for horticultural/agricultural products, including eco-friendly alternatives, if any.

5.2 Marking

5.2.1 Each packet shall be marked in a legible, indelible, and durable as per the provisions of the relevant Indian Standard to give the following information:

- a) Identity of product (namely, dry compost)
- b) Intended use as either soil nutrient or soil conditioner
- c) Name and address of manufacturer
- d) Batch number or code number
- e) Date of manufacture
- f) Date of expiry (agreed between manufacturer and purchaser, subject to minimum 3 months from date of manufacture)
- h) Net quantity
- i) Any other information required under the Legal Metrology (Packaged Commodities) Rules, 2011

5.2.2 Items of **5.2.1 (b), (e), and (f)** shall be printed on a coloured ink background.

5.2.3 Directions for the use of treated faecal sludge compost shall be printed briefly on the packet. A separate pamphlet may preferably be given with it.

5.2.4 Certification Marking

Product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the certification agency's policies and product(s) may be marked with the appropriate certification mark.

5.3 Storage

Treated faecal sludge compost shall be stored by the manufacturer in a cool and dry place away from direct sun or heat. It shall also be the duty of the manufacturer to instruct retailers and users about precautions to be taken during storage.

6. EARLY ADOPTERS

The demand for organic compost has highlighted potential major players in the market.

6.1 Tree farms and pulp and paper industry

Demand for paper in the Indian market has increased due to increasing demand for paper to support the growing education sector. More recently, a ban on single-use plastic products has triggered a demand for paper bags and other paper products. To meet this demand, further growth in the tree farm industry in India is expected in the near future.

6.2 Municipalities

The green highways policy (plantation, transplantation, beautification, and maintenance), approved in 2015, aims to promote greening of highway corridors with participation of community, farmers, private sector, NGOs, and government institutions. With more than 1,430 municipalities governing a network of 6,371,847 km long road, municipalities can be considered major partners as they are responsible for plantation and greening along the road network. Offering a sustainable source of compost will support the government agenda to reduce CO₂ emissions.

Table 1: Testing Specifications for Treated Faecal Sludge Compost

Parameter	Limits	Units	Reference method
Total N*	≥ 0.8	% by weight	WHO 1978
Total P*	≥ 0.8	% by weight	WHO 1978
Total K*	≥ 0.8	% by weight	WHO 1978
Total Organic C	≥ 12	% by weight	GOI 2011
C:N Ratio*	≤ 20:1	~	WHO 1978
Moisture	15–25	% by weight	GOI 2011
pH (Water Extract)	6.5–7.5	1:5 solution	Sullivan et al. 2018
Bulk Density	0.7–0.9	g/cu cm	Agriculture Ministry Methods Manual 2011
Size Distribution	≥ 90%: ≤ 4 mm	mm	IAPMO India internal protocol
Odour	No perceptible	~	IS5401: 2002
Faecal Coliforms	malodour	CFU/g	WHO 1978
Arsenic	200	mg/kg	WHO 1978
Chromium	≤ 10	mg/kg	WHO 1978
Lead	≤ 50	mg/kg	WHO 1978
Nickel	≤ 100	mg/kg	WHO 1978
Mercury	≤ 0.15	mg/kg	WHO 1978

*For use of TFSC as soil conditioner, NPK values need not be measured.

Assessment Requirements for Certification

Prior to certification, assessment shall be conducted of all the crucial parameters specified as in Table 1. The values of these parameters shall be determined by methods as in Annex B. For use as soil conditioner, NPK values need not be measured.

Assessment Requirements for Routine Monitoring

For routine monitoring, assessment shall be conducted at least once every month of the following crucial parameters: CN ratio, moisture, pH, bulk density, stability indicator, inorganic physical contaminants, size distribution, and faecal coliforms. The values of these parameters shall be determined by methods as in Annex B

ANNEX A: SAMPLING OF TREATED FAECAL SLUDGE COMPOST

In drawing, preparing, and handling the samples, the following precautions and directions shall be observed.

- Sampling shall be carried out by a trained and experienced person as it is essential that the sample are representative of the lot to be examined.
- Since the samples are also required for microbiological analysis, utmost care is necessary to avoid extraneous contamination while drawing and handling the samples and to preserve them in their original condition till they are ready for examination in the laboratory.
- No preservative or bactericidal/fungicidal agent shall be added to samples required for microbiological analysis.
- Samples in their original unopened packets shall be drawn and sent to the laboratory. This will prevent possible contamination of the samples during handling and help in revealing the true condition of the material.
- Intact packets shall be drawn from a protected place not exposed to dampness, air, bright light, dust, or soot and transferred to clean containers.
- At least one representative individual or composite sample shall be sent to the laboratory for testing.

Frequency of Sampling

Minimum frequency of drawing representative samples of treated faecal sludge compost for testing parameters specified as in Table 1 shall be at least once every six months for detailed analytical testing and at least once every month for routine testing.

ANNEX B: TESTING OF TREATED FAECAL SLUDGE COMPOST

Methods for Testing Treated Faecal Sludge Compost Parameters listed as in Table 1

Total N (WHO. 1978. Section 2.7. Kjeldahl-Nitrogen. P. 26)

Definition

The Kjeldahl-nitrogen is defined as the nitrogen present in ammonia plus the portion of nitrogen which can be catalytically reduced to ammonia in a concentrated sulfuric acid solution. Kjeldahl-nitrogen generally includes most nitrogen containing compounds like ammonia and organic nitrogen. Nitrates and nitrites are sometimes not completely comprised.

Principle

In a concentrated sulfuric acid solution, the nitrogen of organic compounds can be catalytically reduced. The resulting ammonia is determined by titration

Procedure

Place about 500 mg of the analytical sample to the accuracy of 1 mg into a digestion flask. Add 10 ml conc. sulfuric acid (H₂SO₄, p.a., d = 1.84) and about 1 g catalyst (1 part selen p.a., 2 parts CuSO₄ • 5 H₂O p.a., and 74 parts K₂SO₄ p.a.) or Kjeldahl-tablets according to Wieniger, Merck Nr. 10958. The sample is heated to 300°C for 3-6 hours. Allow the digested sample to cool, add 30-50 ml of distilled water and transfer it to a Kjeldahl distillation apparatus. Add sodium hydroxyde (NaOH p.a. 32 %) until the solution becomes very alkaline. By distillation, the produced ammonia is transferred into a receiver which contains 20 ml 0.05 N sulfuric acid (dilute 1.41 ml conc. H₂SO₄ p.a. with distilled water to 1000 ml). The excess sulfuric acid is titrated with 0.05 N sodium hydroxyde (dilute 2.0 g NaOH p.a. with distilled water to 1000 ml) using the Kolthof indicator (200 mg methyl red and 125 mg methylene blue dissolved in 100 ml ethanol p.a. 96 %). 1 ml 0.05 N sulfuric acid is equivalent to 0.70035 mg nitrogen.

$$N \text{ in \% of the dry residue} = (S-L) \cdot 70.035 / E (+0.05)$$

N = nitrogen content [%]

E = weight of the sample [mg]

S = amount of 0.05 N sulfuric acid in the receiver [ml]

L = amount of 0.05 N sodium hydroxyde used for titration [ml]

Notes

- The nitrogen of some organic compounds cannot be detected by Kjeldahl's method (e.g. amines, azocompounds, etc.).
- By adding a few drops of hydrogen peroxide (H₂O₂ p.a. 30 %) in the final stage of the digestion, the dissolution of hardly digestible substances can be improved.
- Caution: Make sure that the sample is cold when the peroxide is added.

Total P (WHO. 1978. Section 2.11 Phosphorus. Pp. 32-33)

Definition

The phosphorus content is defined as the total amount of phosphorus in a sample expressed as weight percentage

Principle

The sample is digested by a strong oxidant and the dissolved phosphorus is measured photometrically as vanadophospho-ammonium molybdate ($\text{PO}_4 (\text{NH}_4)_3 \text{VdO}_3 \text{NH}_4 16 \text{MoO}_3$).

Procedure

Digestion

Place about 100-250 mg of the analytical sample to the accuracy of 1 mg into an Erlenmayer flask, and add 5 ml of digestion reagent (2 parts of conc. HNO_3 p.a. and 1 part of conc. HClO_4 p.a.). Heat it over a laboratory sandbath until brown nitrogen peroxide and white perchloric acid evaporates. Before complete dryness repeat the procedure until only a white residue remains in the flask.

Colorimetric Measurement

Add 40 ml of vanadium molybdenum* solution to the cold, digested sample, stir the yellow solution for about 1 hour and filter through a G3 glassfilter into a volumetric flask. Rinse the filter with vanadium molybdenum solution until the flask contains exactly 50 ml. Measure the extinction of the clear solution in a 1 cm cell with a spectrophotometer at a wavelength of 470 nm. The phosphorus concentration can be determined by using a calibration curve with potassium dihydrogen-phosphate (KH_2PO_4 p.a.). 1000 mg KH_2PO_4 correspond to 227.6 mg phosphorus. The result is reported in percent phosphorus of the sample.

$P \text{ in } \% = 5 \cdot a/b$

a = conc. of phosphorus in the vanadium molybdenum solution [mg/l]

b = sample weight [mg]

* The three solutions a, b, and c are added in the following order, and the mixture is diluted with distilled water to 1000 ml.

- 67 ml conc. HNO_3 p.a. plus 33 ml distilled water
- Ammonium vanadate 0.25 %: Dissolve 0.25 g NH_4VO_3 p.a. in hot distilled water plus 2 ml conc. HNO_3 p.a., dilute to 100 ml.
- Ammonium molybdate 5 %: Dissolve 5 g (NH_4) $\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$ p.a. in hot distilled water and dilute to 100 ml

Notes

- Between 400-490 nm, the sensitivity of the colorimetric measurement increases with decreasing wavelength.
- Around 400 nm, the absorption is troubled significantly by iron-III.
- Silicates produce the same color as phosphates. The ratio P_2O_5 to SiO_2 should not be less than 1:44.
- The concentration of nitric acid in the solution to be measured should range from 0.2 N and 1.6 N.

Total K (WHO. 1978. Section 2.9. [...] Potassium P. 28-29)

Definition

The content of [...] potassium is defined as the proportion of the corresponding metal in the sample expressed as weight percentage.

Principle

The metals are dissolved in a strong oxidizing reagent. The metal concentration is determined by atomic absorption spectroscopy:

The free metal atoms can absorb specific wave lengths of a continuous spectrum. The amount of light absorbed depends on the concentration of the metal. The free metal atoms are usually produced by vaporizing a solution

Procedure

The method of digestion, AA spectroscopy, and calculations can be applied to the determination of heavy metals, too. It is appropriate to use the same sample for the determination of all metals.

Digestion

Place about 1 g of the analytical sample to the accuracy of 1 mg into an Erlenmeyer flask, and add 5 ml of digestion reagent (2 parts conc. HNO₃ p.a. and 1 part conc. HClO₄ p.a.). Heat it over a laboratory sand-heat until brown nitrogen peroxide and white perchloric acid evaporates. Before the residue is evaporated to dryness, repeat the procedure until only a white precipitant remains in the flask. Then filter through an ash free filter into a 100 ml volumetric flask and dilute the filtrate with 0.1 N nitric acid (p.a.) to 100 ml.

Atomic Absorption Spectroscopy

The concentration of [...] potassium is determined from the 0.1 N nitric acid solution by AA spectroscopy according to the common methods described in AAS manuals.

Calculations

The concentration of the metal X in ppm = $100 \cdot a/b$

a = concentration of X in the solution of the digested sample [mg/1]

b = weight of the sample [g]

Notes

If the concentration of [...] K is very high, the nitric acid solution of the digested sample must be further diluted for atomic absorption spectroscopy.

Total Organic Carbon (GOI 2011 Section 9. Organic Carbon/ Organic Matter Pp. 83-87)

Organic matter estimation in the soil can be done by different methods. Loss of weight on ignition can be used as a direct measure of the organic matter contained in the soil. It can also be expressed as the content of organic carbon in the soil. It is generally assumed that on an average organic matter contains about 58% organic carbon. Organic matter/organic carbon can also be estimated by volumetric and colorimetric methods. However, the use of potassium dichromate ($K_2Cr_2O_7$) involved in these estimations is considered as a limitation because of its hazardous nature. Soil organic matter content can be used as an index of N availability (potential of a soil to supply N to plants) because the content of N in soil organic matter is relatively constant.

Loss of weight on ignition method

Apparatus

- Sieve
- Beaker
- Oven
- Muffle furnace

Procedure

- Weigh 5.0 to 10.0 g (to the nearest 0.01 g) sieved (2 mm) soil into an ashing vessel (50 ml beaker or other suitable vessel).
- Place the ashing vessel with soil into a drying oven set at 105°C and dry for 4 hours. Remove the ashing vessel from the drying oven and place in a dry atmosphere. When cooled, weigh to the nearest 0.01 g. Place the ashing vessel with soil into a muffle furnace and bring the temperature to 400°C. Ash in the furnace for 4 hours. Remove the ashing vessel from the muffle furnace, cool in a dry atmosphere and weigh to the nearest 0.01 g.

Calculation

Percent organic matter (OM) = $(W_1 - W_2)/W_1 \times 100$

Percent organic C = % OM \times 0.58

where,

W_1 is the weight of soil at 105°C and W_2 is the weight of soil at 400°C.

Volumetric method (Walkley and Black, 1934)

Apparatus

- Conical flask - 500 ml
- Pipettes - 2, 10. and 20 ml
- Burette - 50 ml

Reagents

- Phosphoric acid – 85%
- Sodium fluoride solution – 2%
- Sulphuric acid – 96 % containing 1.25% Ag₂SO₄
- Standard 0.1667M K₂Cr₂O₇: Dissolve 49.04 g of K₂Cr₂O₇ in water and dilute to 1 litre
- Standard 0.5M FeSO₄ solution: Dissolve 140 g Ferrous Sulphate in 800 ml water, add 20 ml concentrated H₂SO₄ and make up the volume to 1 litre
- Diphenylamine indicator: Dissolve 0.5 g reagent grade diphenylamine in 20 ml water and 100 ml concentrated H₂SO₄.

Procedure

- Weigh 1.0 g of the prepared soil sample in 500 ml conical flask.
- Add 10 ml of 0.1667M K₂Cr₂O₇ solution and 20 ml concentrated H₂SO₄ containing Ag₂SO₄.
- Mix thoroughly and allow the reaction to complete for 30 minutes.
- Dilute the reaction mixture with 200 ml water and 10 ml H₃PO₄.
- Add 10 ml of NaF solution and 2 ml of diphenylamine indicator.
- Titrate the solution with standard 0.5M FeSO₄ solution to a brilliant green colour.
- A blank without sample is run simultaneously.

Calculation

Percent organic Carbon (X) = $10 (S - T) \times 0.003/S \times 100/\text{Wt. of soil}$

Since one gram of soil is used, this equation simplifies to: $3(S - T)/S$.

where,

S = ml FeSO₄ solution required for blank

T = ml FeSO₄ solution required for soil sample

3 = Eq W of C (weight of C is 12, valency is 4, hence Eq W is $12 \div 4 = 3.0$)

0.003 = weight of C (1,000 ml 0.1667M K₂Cr₂O₇ = 3 g C. Thus, 1 ml 0.1667M K₂Cr₂O₇ = 0.003 g C)

Organic Carbon recovery is estimated to be about 77%. Therefore, actual amount of organic carbon (Y) will be:

Percent value of organic carbon obtained $\times 100/77$

Or Percentage value of organic carbon $\times 1.3$

Percent Organic matter = Y $\times 1.724$ (organic matter contains 58 % organic carbon, hence $100/58 = 1.724$)

Note:

Published organic C to total organic matter conversion factor for surface soils vary from 1.724 to 2.0. A value of 1.724 is commonly used, although whenever possible the appropriate factor be determined experimentally for each type of soil.

Colorimetric method (Datta et al., 1962)

Apparatus

- Spectrophotometer
- Conical flask -100 ml
- Pipettes - 2, 5 and 10 ml

Reagents

- standard potassium dichromate 1/6M (1N)
- Concentrated sulphuric acid containing 1.25% Ag₂SO₄
- Sucrose (AR quality)

Procedure

- Take 1 g of soil in 100 ml conical flask.
- Add 10 ml of 0.1667M K₂Cr₂O₇ and 20 ml of conc. H₂SO₄ containing 1.25 percent of Ag₂SO₄.
- Stir the reaction mixture and allow to stand for 30 minutes. A blank is also prepared in the similar way without adding sucrose.
- The green colour of chromium sulphate so developed is read on a spectrophotometer at 660 nm after setting the blank, prepared in the similar manner, at zero.

Calculation

The carbon content of the sample is found out from the standard curve which shows the carbon content (mg of carbon v/s spectrophotometer readings as absorbance).

Percent C = mg C observed x 100/1000 (observed reading is for 1 g soil, expressed as mg).

Percent OM = %C x 1.724

C:N Ratio (WHO. 1978. Section 2.8. Ratio of Carbon to Nitrogen. P. 27)

Definition

The C/N ratio is the ratio between the calculated or measured carbon and the Kjeldahl-nitrogen.

Principle

A certain C/N ratio is necessary to obtain biological degradation of organic matter. Usually, refuse has a C/N ratio of 30-40 while compost has a ratio of 15-20. The C/N ratio gives some information if a refuse is suited for composting, and indicates the age and maturity of a compost. Also, it is an important criteria for the application of compost in agriculture.

Procedure

Determine carbon content and Kjeldahl-nitrogen. Calculate the C/N ratio, and round off to the closest integer.

$$C/N = \%C/\%N (\pm 1)$$

Moisture (GOI 2011 Section 4. Soil Moisture P. 76)

Gravimetric method of moisture estimation is most widely used where the soil sample is placed in an oven at 105 0C and dried to a constant weight. The difference in weight is considered to be water present in the soil sample.

Apparatus

- Aluminium Moisture Box
- Oven
- Desiccator

Procedure

- Take 100 g of soil sample in the aluminium moisture box and keep in the oven after removing the lid of the box.
- The sample is kept at 105 0C till it attains a constant weight. It may take 24-36 hours.
- Cool the sample, first in the switched off oven and then in a desiccator.
- Take the weight of the cooled moisture box. The loss in weight is equal to moisture contained in 100 g soil sample.

Calculation

$$\text{Moisture(\%)} = \text{Loss in weight/Oven-dry weight of soil} \times 100$$

The corresponding moisture correction factor (mcf) for analytical results or the multiplication factor for the amount of sample to be weighed in for analysis is:

$$\text{Moisture correction factor} = 100 + \% \text{ moisture}/100$$

pH (water extract) (GOI 2011 Section 6. pH P. 77-78)

The soil pH is the negative logarithm of the active hydrogen ion (H^+) conc. in the soil solution. It is the measure of soil sodicity, acidity or neutrality. It is a simple but very important estimation for soils, since soil pH influences to a great extent the availability of nutrients to crops. It also affects microbial population in soils. Most nutrient elements are available in the pH range of 5.5 to 6.5. In various chemical estimations, pH regulation is critical.

Apparatus

- pH meter with a range of 0-14 pH
- Pipette/dispenser
- Beaker
- Glass rod

Reagent

- Buffer solutions of pH 4, 7, and 9
- Calcium chloride solution (0.01M): Dissolve 14.7 g $CaCl_2 \cdot 2H_2O$ in 10 litre of water to obtain 0.01M solution

Procedure

- Calibrate the pH meter, using 2 buffer solutions, one should be the buffer with neutral pH (7.0) and the other should be chosen based on the range of pH in the soil. Take the buffer solution in the beaker. Insert the electrode alternately in the beakers containing 2 buffer solutions and adjust the pH. The instrument indicating pH as per the buffers is ready to test the samples.
- Weigh 10.0g of soil sample into 50 or 100 ml beaker, add 20ml of $CaCl_2$ solution (use water instead of $CaCl_2$ solution throughout the procedure if water is used as a suspension medium).
- Allow the soil to absorb $CaCl_2$ solution without stirring, then thoroughly stir for 10 second using a glass rod.
- Stir the suspension for 30 minutes and record the pH on the calibrated pH meter

Calculations

- Specific colour changes due to pH change in the presence of pH indicators are as adapted from pH Indicators, E. Merck and Co.
- Based on soil pH values of <4.6, 4.6-5.5, 5.6-6.5, 6.6-6.9, 7.0, 7.1-8.5, and >8.5, soil reactions are rated as Extremely acid, Strongly acid, Moderately acid, Slightly acid, Neutral, Moderately alkaline, and Strongly alkaline, respectively. Acidic soils need to be limed before they can be put to normal agricultural production. Alkali soils need to be treated with gypsum to remove the excessive content of sodium

Bulk Density (Sullivan et al. 2018)

Bulk density is expressed in pounds per cubic yard. Bulk density testing can be done in a laboratory or in the field. Field determination of bulk density often is more informative than laboratory measurement because it uses a larger volume of compost. As a rule of thumb, screened composts that contain 50 percent moisture will have a bulk density of about 1,000 lb/cu yd. Very wet composts can have a bulk density of over 1,500 lb/cu yd. Multiple samples can be evaluated to obtain an average bulk density.

“As-is” bulk density is the weight of compost present per unit volume. When applying compost by volume (e.g., spreader load), estimates of as-is compost bulk density and moisture are needed to estimate dry matter application rates. Depending on moisture content, particle size, and compaction, bulk density can range from 800 to more than 1,600 lb/cu yd.

Procedure

As-is bulk density can be measured on-site, using a scale and a 5-gallon bucket with vertical sides, as follows:

- Weigh the empty bucket and record its weight.
- Fill the bucket with 5 gallons of water and mark the water level on the inside of the bucket. Empty the bucket. Use a ruler to measure and mark lines at one-third and two-thirds of the 5-gallon line.
- Fill the bucket with compost to the one-third line. Drop the bucket 10 times from a height of 1 foot. Add compost up to the two-thirds line and drop the bucket 10 times. Add compost to the 5-gallon line and drop 10 times. After the final drop, top off the bucket with compost to the 5-gallon line.
- Weigh the full bucket and subtract the weight of the empty bucket. This is the weight of 5 gallons of compost.
- Multiply this weight by 40 to calculate the bulk density in lb/cu yd.
- Repeat 2–3 times and take the average.

Size Distribution (GOI 2011 Section 2. Soil Structure Pp. 72-74)

The determination of aggregate or clod size distribution involves procedures that depend on the disintegration of soil into clods and aggregates. For the measurements to have practical significance, the disruptive forces causing disintegration should closely compare with the forces expected in the field.

Dry Aggregates Analysis (Gupta and Ghil Dyal, 1998)

The size distribution of dry clods is measured by Dry Sieving Analysis performed on air dry bulk soil sample either manually or with the help of a rotary sieve shaker.

Apparatus

- Nest of sieves, 20 cm in diameter and 5 cm in height, with screens having 25.0, 10.0, 5.0, 2.0, 1.0, 0.5 and 0.25 mm size round openings with a pan and a lid
- Rotary sieve shaker
- Aluminium cans
- Balance
- Spade
- Brush
- Polyethelene bags
- Labels

Procedure

- Bulk soil sample is collected from the tilled field with the help of a 20 cm diameter and 10 cm height ring. The ring is placed on the tilled soil and pressed until in level with the surface. The loose soil within the ring is removed and collected in a polyethelene bag.
- One label indicating the depth and soil profile is put inside the bag and the other label is tied with the bag. The soil samples are then brought to the laboratory and air dried.
- Spread the soil on a sheet of paper and prepare the subsamples by 'quartering'. The mixed soil material is coned in the center of the mixing sheet with care to make it symmetrical with respect to fine and coarse soil material. The cone is flattened and divided through the center with a flat metal spatula or metal sheet, onehalf being moved to the side quantitatively. Each one-half is further divided into halves; the four quarters being separated into separate piles or 'quarters'. The subsamples from two of these 'quarters' are weighed and used for clod size and aggregate distribution analysis as duplicates. The weighed soil sample is transferred to the top sieve of the nest of sieves having 5.0, 2.0, 1.0, 0.5 and 0.25 mm diameter round openings and a pan at the bottom. Cover the top sieve with the lid and place the nest of sieves on a rotary shaker. Switch on the shaker for 10 minutes, and then remove the sieves, and collect the soil retained on each screen in the pre-weighed aluminium cans, with the help of a small brush, and weigh the cans with the soil.

- If the percentage of dry aggregates on 5 mm sieve exceeds 25%, transfer these aggregates to a nest of sieves with 25.0, 10.0, and 5.0 mm sieves along with a pan.
- Cover the top sieve containing the aggregates with a lid and place the nest of sieves on the rotary sieve shaker. Switch on the motor for 10 minutes and proceed as above for the estimation of aggregate size distribution. Analyse the duplicate sample following the same procedure and calculate the percent distribution of dry aggregates retained on each sieve.
- Duplicate 100 g sample is dried in an oven for 24 hours at 1050 C to calculate the oven dry weight of the soil sample.

Odour (IAPMO India internal protocol)

Treated faecal sludge compost shall be free of perceptible malodour or objectionable smell

Procedure:

Odour can be tested by sniffing at a distance of 15 cm to ascertain that malodour is absent.

Faecal Coliforms (IS 5401: 2002)

Most human pathogens occur from faecal matter and all faecal matter is loaded in faecal coliforms. Faecal coliforms can survive in both aerobic and anaerobic conditions and are common in all initial compost piles. Therefore, faecal coliforms are used as an indicator to determine if the chosen method for pathogen reduction (heat for compost) has met the requirements of sufficient temperature, time and mixing. If faecal coliforms are <1000 / g dry weight, it can be assumed that all others pathogens are eliminated.

Potential problems are that faecal coliforms can regrow during the curing phase or during shipping. This is because the conditions are now more favourable for growth than during the composting process.

Definition

Coliforms are defined as bacteria which, at the specified temperature (i.e. 30 °C, 35 °C or 37 °C, as agreed) form characteristic colonies in crystal violet, neutral red, bile lactose agar under the test conditions specified in this Standard.

Culture medium and dilution fluid

General

For current laboratory practice, see ISO 7218.

Dilution fluid

See ISO 6887 and the specific International Standard dealing with the product under examination.

Solid selective medium: crystal violet, neutral red, bile lactose (VRBL) agar

- Composition
- peptone 7g, yeast extract 3g, lactose 10 g, sodium chloride 5 g, bile salts 1.5 g, neutral red 0.03 g, crystal violet 0.002 g, agar 12 g to 18 g (according to gel strength of agar), water 1000ml
- Preparation (to conserve the selective power and specificity of the medium)
- Thoroughly mix the components or the dehydrated complete medium in the water and leave to stand for several minutes. Adjust the pH so that, after boiling, it is 7.4 at 25 °C. Bring to the boil, stirring from time to time.
- Allow to boil for 2 min. Immediately cool the medium in the water-bath set at 45 °C.
- Avoid overheating the medium, heating it for too long or reheating it. Consequently, do not sterilize in the autoclave, and check the sterility of the medium at the time of use.
- Use the medium within 3 h of its preparation.

Apparatus and glassware

- Apparatus for dry sterilization (oven) or wet sterilization (autoclave) See ISO 7218.
- Incubator capable of operating at $30\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$, $35\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$ or $37\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$.
- Petri dishes made of glass or plastic, of diameter 90 mm to 100 mm.
- Total delivery pipettes having a nominal capacity of 1 ml.
- Water-bath, or similar apparatus capable of operating at $45\text{ }^{\circ}\text{C} \pm 0.5\text{ }^{\circ}\text{C}$
- Colony counting equipment, consisting of an illuminated base and a mechanical or electronic digital counter.
- pH meter, accurate to ± 0.1 pH unit at $25\text{ }^{\circ}\text{C}$.
- Disposable apparatus is an acceptable alternative to reusable glassware if it has suitable specifications.

Sampling

Sampling shall have been carried out in accordance with the specific International Standard appropriate to the product concerned. If there is no specific international Standard, it is recommended that the parties concerned come to an agreement on this subject.

Procedure

Test portion, Initial suspension, and dilutions

- See ISO 6887 and the specific International Standard appropriate to the product concerned.

Inoculation and Incubation

- Take two sterile Petri dishes. Using a sterile pipette, transfer to each dish 1 ml of the test sample, if the product is liquid, or 1 ml of the initial suspension In the case of other products.
- Take two other sterile Petri dishes. Using a fresh sterile pipette, transfer to each dish 1 ml of the first decimal dilution (10⁻¹) of the test sample, if the product is liquid, or 1 ml of the first decimal dilution (10⁻²) of the Initial suspension in the case of other products.
- Repeat the procedure described with the further dilutions, using a fresh sterile pipette for each decimal dilution.
- Pour about 15 ml of the VRBL medium, at $45\text{ }^{\circ}\text{C} \pm 0.5\text{ }^{\circ}\text{C}$, into each Petri dish. The time elapsing between the end of the preparation of the initial suspension (or of the 10⁻¹ dilution if the product is liquid) and the moment when the medium is poured into the dishes shall not exceed 15 min.
- Carefully mix the inoculum with the medium and allow the mixture to solidify, with the Petri dishes standing on a cool horizontal surface.
- Also prepare a control plate, with 15 ml of the medium for checking its sterility.
- After complete solidification, pour about 4 ml of the VRBL medium (5.3), at $45\text{ }^{\circ}\text{C} \pm 0.5\text{ }^{\circ}\text{C}$, on to the surface of the inoculated medium. Allow to solidify as described above.
- Invert the prepared dishes and incubate them in the incubator set at $30\text{ }^{\circ}\text{C}$, $35\text{ }^{\circ}\text{C}$ or $37\text{ }^{\circ}\text{C}$ (as agreed) for 24 h f 2 h.

Counting of the colonies

- After the specified period of incubation, count, using the colony counting equipment, the characteristic coliform colonies in each dish containing not more than 150 colonies, whether characteristic or not.
- Note: After incubation for 24 h, characteristic colonies are purplish red colonies having a diameter of 0.5 mm or greater and sometimes surrounded by a reddish zone of precipitated bile

Heavy metals (Arsenic, Chromium, Lead, Nickel, Mercury) (WHO. 1978. Section 2.10. heavy Metals. Pp. 30-31)

Definition

- Heavy metals is the term applied to those metallic elements which have a density >6 g/cm. In the context of refuse and compost, the most important heavy metals are: iron, cobalt, nickel, copper, zinc, lead, cadmium, chromium, manganese, and mercury.
- The concentration of heavy metals is reported in ppm, or as percentage if the concentration is very high (iron).

Principle

- The metals are dissolved in a strong oxidizing reagent. Their concentration is determined by atomic absorption spectroscopy (AAS) or inductively coupled plasma optical emission spectrometry (ICPOES) or inductively coupled plasma mass spectrometry (ICPMS).
- Mercury can not be determined with this method alone; the procedure of this method needs to be coupled with hydride vapour generation technique

Procedure

The methods of digestion, AA spectroscopy, and calculations are the same as those used for Potassium. It is appropriate to use the same sample for the determination of all metals.

Digestion

- Place about 1 g of the analytical sample to the accuracy of 1 mg into an Erlenmeyer flask, and add 5 ml of digestion reagent (2 parts conc. HNO_3 p.a. and 1 part conc. HClO_4 p.a.). Heat it over a laboratory sand-heat until brown nitrogen peroxide and white perchloric acid evaporates. Before the residue is evaporated to dryness, repeat the procedure until only a white precipitant remains in the flask. Then filter through an ash free filter into a 100 ml volumetric flask and dilute the filtrate with 0.1 N nitric acid (p.a.) to 100 ml.
- After extraction step of this method is completed, mercury can be analysed by ICPOES with hydride generator.

Atomic Absorption

- The concentration [...] is determined from the 0.1 N nitric acid solution by AA spectroscopy according to the common methods described in AAS manuals

Calculations

- The concentration of the metal X in ppm = $100 \cdot a/b$
- a = concentration of X in the solution of the digested sample [mg/l]
- b = weight of the sample [g]

Notes

- This digestion method is not suited for samples to be analyzed by atomic absorption using a graphite tube.
- If dealing with very low concentrations, the digested sample has to be diluted with less than 100 ml 0.1 N nitric acid.

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IAPMO – I TFSC – 01: 2022

Developed by



In collaboration with



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