

Report Summary

CLIENT: LEETHA INDUSTRIES PRIVATE LIMITED.

SAMPLE REGISTRATION DATE: 15/09/2018

TESTING COMPLETION DATE: 30/04/2019

SAMPLE DESCRIPTION: COATED PAPER CUP

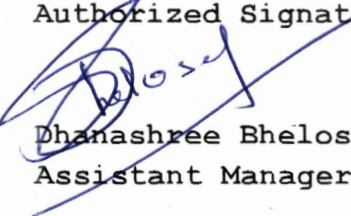
QUANTITY RECEIVED: 1 PACKET

TEST CONDUCTED: ASTM D6400: STANDARD TEST METHOD FOR DETERMINING AEROBIC BIODEGRADATION OF PLASTIC MATERIALS UNDER HIGH-SOLIDS AEROBIC-DIGESTION CONDITIONS CONSIDERING GAS EVOLUTION

REPORT NO.: MUM/003382B/2018

COATED PAPER CUP SAMPLE SUBMITTED BY **LEETHA INDUSTRIES PRIVATE LIMITED** SHOWS 91.85% BIODEGRADATION AT 75TH DAY IN ACCORDANCE TO THE CONDITIONS SET FORTH IN ASTM D 6400.

Authorized Signatory


Dhanashree Bhelose
Assistant Manager - Environment



REPORT LEETHA

**ASTM 6400
LABELING OF PLASTICS DESIGNED TO BE AEROBICALLY
COMPOSTED IN MUNICIPAL OR INDUSTRIAL FACILITIES**

ENVIRONMENTAL DIVISION LABORATORY, MUMBAI

INTERTEK INDIA PRIVATE LIMITED

Client: LEETHA INDUSTRIES PRIVATE LIMITED.

Sample registration date: 15/09/2018

Analysis starting date: 08/01/2019

Analysis completed on: 30/04/2019

Name of product: Coated Paper cup

Quantity received and packing: - 1 packet

Sample details: Coated Paper cup

Coating Specifications - M/s.Leetha Group of Industries developed an aqueous dispersion. It is **U.S. FDA 175.105 & 175.300** approved & can be used in **Direct Food Contact** & can also be coated on Papers to make the Paper Heat sealable, Oil & water resistance. Same coating is coated on Paper Board used for making paper cups & marked as "Coated Paper Cup".

Test Required: IS/ISO 17088 Specifications for compostable plastics / ASTM D6400 Labeling of plastics designed to be aerobically composted in municipal or industrial facilities

Sampling done by: Sample not drawn by Intertek

Report No. MUM/003382B/2018

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LABORATORY

Testing as presented in this report was conducted by Environmental division of Intertek India Private Limited. The testing facility is located at F wing, 2nd Floor, Chandivali Saki vihar Road, Andheri (East), Mumbai – 400 072, India.

SAMPLE RECEIPT

The sample was received on 15th September 2018 at the Intertek testing facility. The sample was sent through courier. Sample was at ambient temperature in good condition with no evidence of damage or contamination. No temperature preservation was required.

SAMPLE DESCRIPTION:



Figure 1: Paper Cup

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PROJECT DESCRIPTION:

Paper cup samples were submitted by LEETHA Industries for testing under **standard IS/ISO 17088 & ASTM D 6400**. The purpose of this specification is to establish requirements for identifying items made from plastics or polymers so that they do not interfere with their satisfactorily composting in commercial and municipal aerobic composting facilities. Products meeting the requirements outlined below be labeled as “compostable in municipal or industrial aerobic facilities”

In order to compost satisfactorily, a product or material must demonstrate each of the characteristics given below

- **Disintegration During Composting**—A plastic product or material will disintegrate during composting such that any remaining plastic residuals are not readily distinguishable from the other organic materials in the finished product. Additionally, the material or product must not be found insignificant quantities during screening prior to final distribution of the compost.
- **Biodegradation**—A level of biodegradation for the plastic products shall be established by tests under controlled conditions.
- **No Adverse Impacts on Ability of Compost to Support Plant Growth**—The tested materials shall not adversely impact on the ability of composts to support plant growth, when compared to composts derived from bio waste without any addition of tested products or reference materials. Additionally, the polymeric products or materials must not introduce unacceptable levels of regulated metals or hazardous substances into the environment, upon sample decomposition.

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Detailed Requirements

- **Disintegration during Composting**—A plastic product is considered to have demonstrated satisfactory disintegration if after twelve weeks (84 days) in a controlled composting test, no more than 10 % of its original dry weight remains after sieving on a 2.0-mm sieve. The test shall be carried out in accordance with ASTM D5338 under thermophilic composting conditions.
- **Ultimate Aerobic Biodegradation**—A plastic product must demonstrate a satisfactory rate and level of biodegradation by achieving the following ratio of conversion to carbon dioxide given below(a) within 180 days using Test Method D5338
 - For all polymers, Ninety percent (90 %) of the organic carbon shall be converted to carbon dioxide by the end of the test period when compared to the positive control. Both the positive control, and test sample to be composted for the same length of time
- A plastic product can demonstrate satisfactory terrestrial safety if it fulfills the requirements below
 - The plastic or product shall have concentrations of regulated metals less than 50 % of those prescribed for sludge's or composts in the country where the product will be placed on the market or disposed of.
 - The germination rate and the plant biomass of the sample composts shall be no less than 90% that of the corresponding blank composts for two different plant species following OECD Guideline 208

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ASTM D5338:**Principle of Biodegradation:**

Biodegradability of the entire fragmented product from Tier 1 under lab-scale conditions is done using Test methods ASTM D5338. This method determines the degree and rate of aerobic biodegradation of plastic materials on exposure to a controlled composting environment under laboratory conditions, at thermophilic temperatures. The samples were exposed to an inoculum that is derived from compost from municipal solid waste. The aerobic composting takes place in an environment where temperature, aeration and humidity are closely monitored and controlled. The percentage of biodegradability is obtained by determining the percentage of carbon in the test sample that is converted into CO₂ during the duration of the test.

Apparatus Setup:

A series of 09 composting vessels of 5-liter volume (1 blank i.e. compost, 1 positive i.e. cellulose mixed with compost, and 1 test sample mixed with compost, all in 3 replicates). The entire composting vessels were kept in Incubator capable of maintaining the temperature of composting vessels at 58± 2° C (Figure 3). Pressurized air system that provides CO₂ free, H₂O saturated air to each of the composting vessels at accurate aeration rate. CO₂ evolved will be absorbed by 0.024 N Ba (OH)₂ and the amount of CO₂ will be determined by titrating with 0.05 N HCl.

Compost Inoculum

The compost inoculum should be 2 to 4 months old, well aerated compost from organic fraction of municipal solid waste, sieved on a screen of less than 10 mm, compost from plants, treating green or yard waste or mixtures of municipal solid waste and green waste should be used. The compost inoculum should produce 50-150 mg of CO₂ per gram of volatile solids over the first 10 days of the test and an ash content of less than 70% and a pH between 7 and 8.2, is desired. The amount of total dry solids may range from 50 to 55%. Compost should have enough

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porosity to enable aerobic conditions. The compost inoculum should be as free from larger inert materials (glass, stones, metals, etc.) as possible.



Figure 2: Compost inoculum

PROCEDURE

The composting vessels will be incubated in diffuse light minimum for a period of 45 days or more & the temperature of the system will be maintained at 58°C. The CO₂ & O₂ concentrations will be checked in the outgoing air. The air flow will be adjusted to maintain a CO₂ concentration of at least 2% vol /vol to allow accurate determination of CO₂ level in the exhaust air. Composting vessels will be shaken weekly to prevent extensive channeling provide uniform attack on test specimen and provide an even distribution of moisture. The incubation time will be fixed to a minimum 45 days.

Carbon Dioxide Analysis:

The carbon dioxide (CO₂) produced in each vessel reacted with Ba (OH)₂ and will be precipitated as barium carbonate (BaCO₃). The amount of carbon dioxide produced will be determined by titrating the remaining barium hydroxide with 0.05 N hydrochloric acid to a

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phenolphthalein end point. Data obtained from the titration will be used to calculate the amount of CO₂ produced.

RESULTS:

Disintegration during Composting

Leetha Paper Cup sample has disintegrated and 8% of its original dry weight is retained after sieving on a 2.0-mm sieve

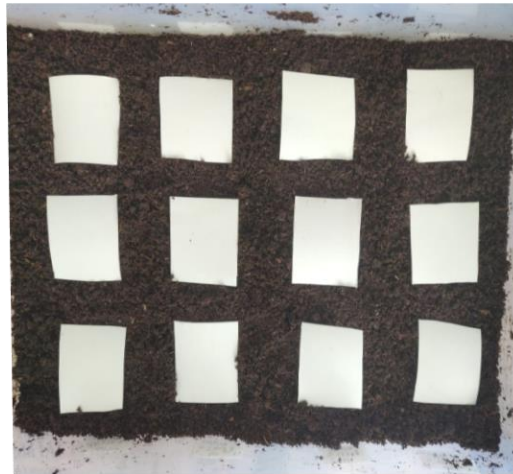


Figure 3: Leetha Paper Cups before composting process.



Figure 4: Leetha Paper Cups After composting process.

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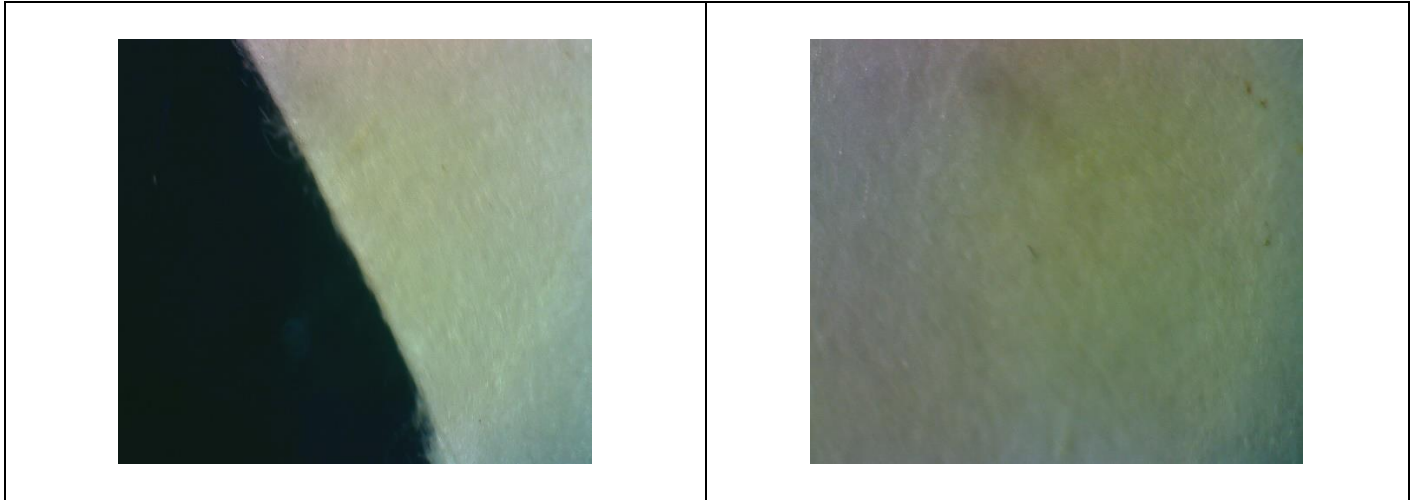


Figure 5: Microscopic view of Paper Cup Before composting process.

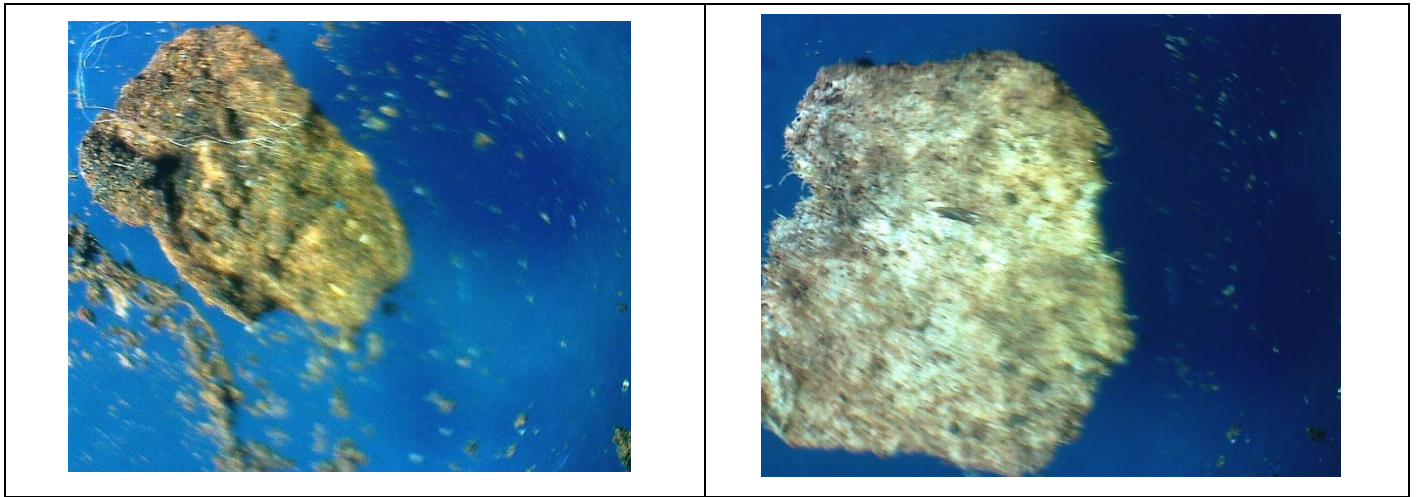


Figure 6: Microscopic view of Paper Cup After composting process

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Ultimate Aerobic Biodegradation

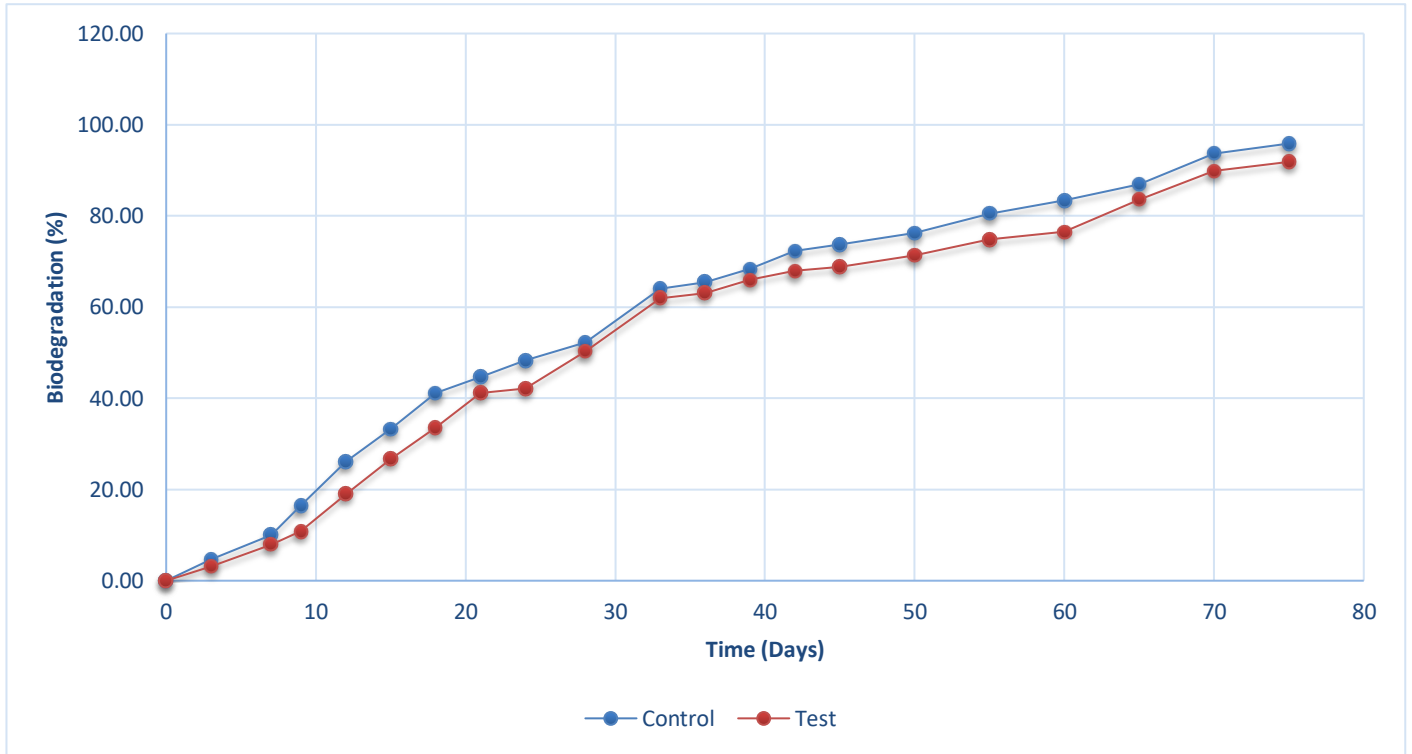
The Paper Cup samples were subjected to biodegradation as per ASTM D5388-15: biodegradability under controlled composting conditions at 58°C ± 2 °C and biodegradability was determined by measuring the actual metabolic conversion of the compostable material into carbon dioxide using the standard test method.

After 75 days of incubation under dry (58 °C ± 2 °C), aerobic controlled composting conditions using test method ASTM D 5338-15, the reference (Positive control), and **Paper Cup** sample were gradually biodegraded. The reference sample was degraded more than 95.87 % after 75 days while the Leetha Paper Cup sample showed only 91.85% after 75 days (Graph 1)

Table 1: Percentage Biodegradation of Positive control and Test samples

Day	% Biodegradation	
	Positive Control	Paper Cup
0	0.00	0.00
3	4.65	3.13
7	10.02	7.96
9	16.46	10.81
12	26.11	19.05
15	33.27	26.73
18	41.14	33.55
21	44.72	41.23
24	48.29	42.09
28	52.23	50.33
33	64.03	61.99
36	65.46	63.13
39	68.32	65.97
42	72.26	67.96
45	73.69	68.82
50	76.19	71.37
55	80.49	74.79
60	83.35	76.49
65	86.93	84.00
70	93.72	89.86
75	95.87	91.85

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Graph 1: Percentage biodegradation of Leetha Paper Cup under aerobic composting

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Eco- toxicity :

Phytotoxicity test by OECD 208 involves the assessment of the seedling Emergence and seedling Growth of higher plants following the exposure to the test substance in the soil and OECD 207 which involves earth worm toxicity test. It is used to confirm that biodegraded and composted materials do not introduce toxic components into the compost.

Principle of OECD 208:

Seeds of Brassica Juncea and Triticum aestivum are placed in contact with soil treated with the test substance and evaluated for effects following 14 to 21 days after 50% emergence of the seedlings in the control group. Endpoints measured are visual assessment of seedling emergence, biomass (fresh or dry shoot weight, or shoot height) and visual detrimental effects (chlorosis, mortality, plant development abnormalities, etc.). Measurements are made at least weekly or more often when recording the emergence of the seeds and compared to those of untreated control plants.

In order for the test to be considered valid, the following performance criteria must be met in the controls:

- the seedling emergence should be at least 80% for crop and 65 % for non-crop species;
- the mean control seedling growth does not exhibit visible phytotoxic effects (e.g. chlorosis, necrosis, wilting, leaf and stem deformation);
- the mean control survival is at least 90% for the duration of the study;
- for any species, all organisms in a test must be from the same source;
- all test chambers or rooms used for a particular species should be identical and should have Same conditions and contain same amount of soil matrix, support media, or substrate from the same source.

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Principle of OECD 207:

Similarly, *Eisenia foetida* species of earthworm are used for earthworm toxicity testing. The artificial soil test involves keeping earthworms in samples of a precisely defined artificial soil to which a range of concentrations of the test substance has been applied. Mortality is assessed 7 and 14 days after application. Control are used to assure that effects observed are associated with or attributed only to the test substance exposure. The earthworm is brought from Vermicomposting beds.

The mortality in the controls should not exceed 10 per cent at the end of either test.

RESULTS:

Metal analysis of compost of test Paper Cup Sample was done to check the toxic levels after biodegradation (Table 3). Seeds grown in compost prepared from samples showed more than 90% germination rate as compared to control. Root and shoot length of plant was similar or slightly more than controls (Table 4). There was no visual injury found in the roots and shoot of the plant due to the test substance.

After 7 days and 14 days, 100% earthworm survival was observed in both 50% and 25% concentration of compost (Figure 7).

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Table 2: Metal analysis of residue collected after biodegradation

PARAMETERS	UNIT	Paper Cup Sample
pH	-	7.82
Mg	PPM	7509
Ca	PPM	13210
P	PPM	864
Mn	PPM	910
Cu	PPM	122
Zn	PPM	122
Ni	PPM	0.67
Cd	PPM	<1.00
Pb	PPM	1.23
Hg	PPM	<1.00
Se	PPM	<1.00
As	PPM	<1.00

As per the Environmental Protection Agency (EPA) the metal concentrations in the sample are well within the prescribed limit

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Table 3: Germination Rate and Biomass of Brassica Juncea and Triticum aestivum seeds after 21 days.

	Plant species	Dose	Germination Rate (%)	Shoot Length (cm)	Root Length (cm)
Control	<i>Brassica juncea</i>	25%	98	10.1	4.3
	<i>Brassica juncea</i>	50%	99	10.6	4.2
	<i>Triticum aestivum</i>	25%	99	16.3	15.1
	<i>Triticum aestivum</i>	50%	98	16.1	15.2

	Plant species	Dose	Germination Rate (%)	Shoot Length (cm)	Root Length (cm)
Paper Cup Sample	<i>Brassica juncea</i>	25%	99	10.2	4.5
	<i>Brassica juncea</i>	50%	97	10.0	4.0
	<i>Triticum aestivum</i>	25%	98	15.8	14.8
	<i>Triticum aestivum</i>	50%	97	15.5	14.7

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The above study was conducted at 25°C ± 4 °C temperature, 65% ± 10% humidity and 55.74 FC light intensity with 16 hours of light. The above results showed that the **Paper Cup Sample** showed no effect on the plant growth and showed no visible damage to the plants.

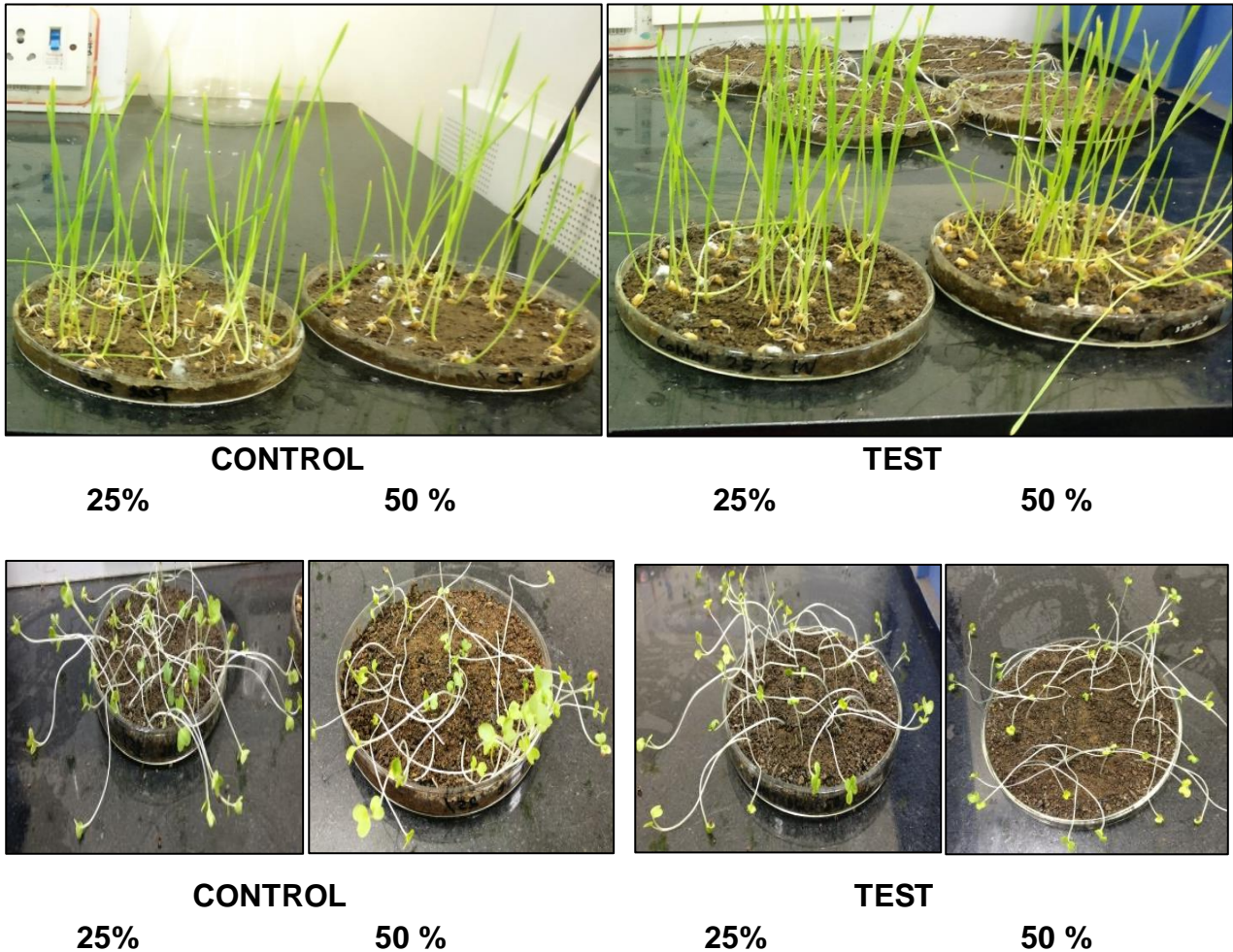


Figure 7: Effect of compost containing Paper Cup Sample film sample on *Triticum aestivum* and *Brassica Juncea* growth

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Earthworm Toxicity

The earthworm toxicity study was conducted as below-

Artificial soil was prepared using 10% sphagnum peat, 20% kaolin clay and 70% industrial sand with final pH 6.32, moisture content 37.8%. the study was conducted at 20°C± 2 °C in continuous light with light intensity approximately 580 Lux.

Average weight of 10 earthworms before and after study

Table 4: Average weight of earthworms before and after study

Initial

Particulars	SET 1		SET 2	
	25%	50 %	25%	50%
Control (g)	0.5528	0.5573	0.6101	0.6221
Paper Cup Sample(g)	0.6013	0.5569	0.5998	0.5986

Final

Particulars	SET 1		SET 2	
	25%	50 %	25%	50%
Control (g)	0.5994	0.6723	0.6550	0.6520
Paper Cup Sample(g)	0.6120	0.6210	0.6542	0.6487

The mortality rate was found to be 0%. The average weight of earthworms in increased as compared to control in some cases.

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BEFORE

AFTER

Figure 8: Earthworm before and after exposing it to the compost containing Paper Cup residue

CONCLUSION:

In accordance to the conditions set forth in ASTM D6400 & IS/ISO 17088, Paper Cup samples submitted by Leetha disintegrated after 75 days exposure and showed 91.85% organic carbon was converted to carbon dioxide relative to the positive control. Toxicity test was conducted as per OECD 207&208 on compost Paper Cup residue. The Paper Cup did not show any effect on the seedling emergence, seedling growth and passed the earthworm toxicity test.

Authorized Signatory

Dhanashree Bhelose
Assistant Manager – Environment

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