

Report Summary

CLIENT: LEETHA INDUSTRIES PRIVATE LIMITED.

SAMPLE REGISTRATION DATE: 15/09/2018

TESTING COMPLETION DATE: 05/01/2019

SAMPLE DESCRIPTION: COATED PAPER CUP

QUANTITY RECEIVED: 1 PACKET

TEST CONDUCTED: ASTM D5511: STANDARD TEST METHOD FOR DETERMINING ANAEROBIC BIODEGRADATION OF PLASTIC MATERIALS UNDER HIGH-SOLIDS ANAEROBIC-DIGESTION CONDITIONS CONSIDERING GAS EVOLUTION

REPORT NO.: MUM/003382/2018

COATED PAPER CUP SAMPLE SUBMITTED BY **LEETHA INDUSTRIES PRIVATE LIMITED** SHOWS 82.41% BIODEGRADATION AT 52ND DAY IN ACCORDANCE TO THE CONDITIONS SET FORTH IN ASTM D 5511.

ASTM D 5511 STATES THAT FOR THE TEST TO BE CONSIDERED VALID, THE POSITIVE CONTROL (REFERENCE MATERIAL) MUST ACHIEVE 70 % BIODEGRADATION WITHIN 30 DAYS. **REFERENCE MATERIAL (CELLULOSE)** SHOWED 74.8 % BIODEGRADATION ON 28TH DAY AND 94.01% ON 52ND DAY.

Authorized Signatory


Dhanashree Bhelose

Assistant Manager - Environment



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REPORT

ASTM D5511

Standard Test Method for Determining Anaerobic Biodegradation of Plastic Materials under High-Solids Anaerobic-Digestion Conditions Considering Gas Evolution

ENVIRONMENTAL DIVISION LABORATORY, MUMBAI

INTERTEK INDIA PRIVATE LIMITED

Client: LEETHA INDUSTRIES PRIVATE LIMITED.

Sample registration date: 15/09/2018

Analysis starting date: 24/10/2018(pre-conditioning) **Analysis completed on:** 05/01/2019

Name of product: Paper cup

Quantity received and packing:- 1 packet

Sample details: Coated Paper cup

Test Required: ASTM D5511 - Standard Test Method for Determining Anaerobic Biodegradation of Plastic Materials under High-Solids Anaerobic-Digestion Conditions Considering Gas Evolution

Sampling done by: Sample not drawn by Intertek

Report No. MUM/003382/2018

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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

PROJECT DESCRIPTION:

Paper cup samples were submitted by LEETHA Industries for testing under standard ASTM D5511. This test method covers the determination of the degree and rate of anaerobic biodegradation of plastic materials in high-solids anaerobic conditions. The test materials are exposed to a methanogenic inoculum derived from anaerobic digesters operating only on

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pretreated household waste. The anaerobic decomposition takes place under high-solids (more than 30 % total solids) and static non-mixed conditions. This test method is designed to yield a percentage of conversion of carbon in the sample to carbon in the gaseous form under conditions found in high-solids anaerobic digesters, treating municipal solid waste.

INOCULUM COLLECTION AND CONDITIONING

The anaerobic digested sewage sludge (Figure 2) mixed with household waste was obtained from the Chembur (Mumbai). To make the sludge adapted and stabilized during a short post-fermentation at 53 °C, the sludge was pre-incubated (one week) at 53 °C. This means that the concentrated inoculum was not fed but allowed to post ferment the remains of previously added organics. Therefore, large easily biodegradable particles were degraded during this period and reduce the background level of biogas from the inoculums itself.

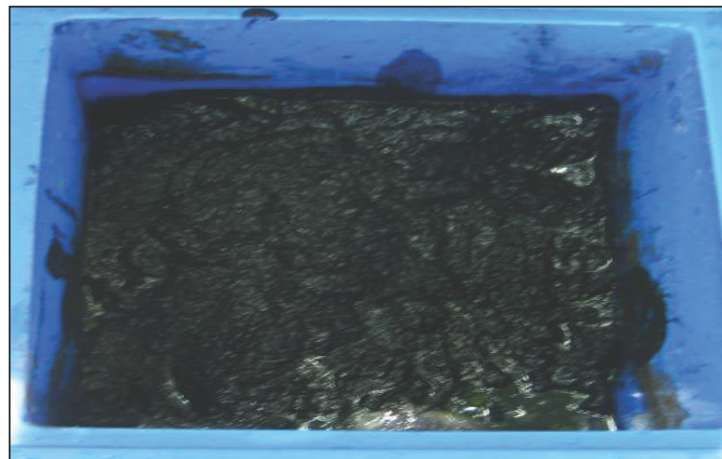


Figure 2: Anaerobic microbial inoculum

INOCULUM PROPERTIES

A sample of the anaerobic digested sewage sludge was analyzed for pH, percent dry solids, and volatile solids, as well as, the amount of CO₂ and CH₄ evolution during the testing. Table 1 lists the results of this initial testing.

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Table 1: Results of initial testing of the anaerobic digested sewage sludge

Parameters	Requirement	Actual results
pH	7.5 to 8.5	7.82
Kjeldahl nitrogen	0.5 and 2 g/kg wet weight	1.00
Dry Solids at 105 °C	20%	26.00

METHODOLOGY:

ASTM D5511 determines the degree of anaerobic biodegradation of plastic materials in high-solids anaerobic conditions. The test sample is exposed to a methanogenic inoculum cultivated from a wastewater treatment facility's anaerobic digesters operating on household waste. After the inoculum is prepared, the samples are placed in incubation vessels and kept within strict temperature and moisture tolerances. The vessels are airtight and are not exposed to UV radiation. Three vessels are used for test, one inoculum, one positive control and one sample. After an initial burst of aerobic activity, the oxygen in the incubation vessels is depleted and the anaerobic bacteria begin to multiply and colonize in the vessel. Many factors affect the biodegradability of polymers, including pH, bacterium type, temperature, molecular weight, chemical linkages, and access of the material to the enzymatic system. As these bacteria begin to utilize the carbon in the test samples, they produce carbonaceous gas such as CH₄ and CO₂ (Figure 2&3). These gases are measured and the results are carefully recorded. If the positive control (which contains cellulose) continues to exhibit biodegradation, then the test is considered valid and the inoculum considered alive. When the test is run to satisfaction, final gas readings are recorded and the incubation vessels are emptied and the samples are cleaned, weighed and the percent biodegradation of the samples can be determined based on conversion of carbon from the test material to carbon in the gaseous phase (CH₄ and CO₂).

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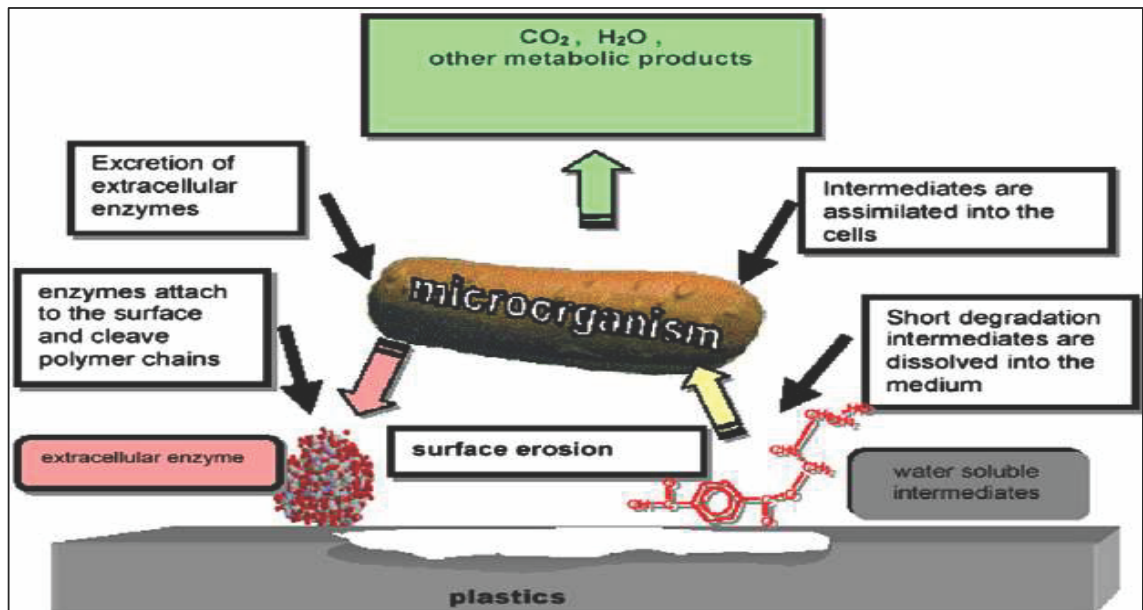


Figure 3: Mechanism - biodegradable plastic

ANAEROBIC DIGESTER SETUP FOR THE PLASTIC BIODEGRADATION

The biodegradation testing of sample was performed in the digester as shown in the (Figure-2).

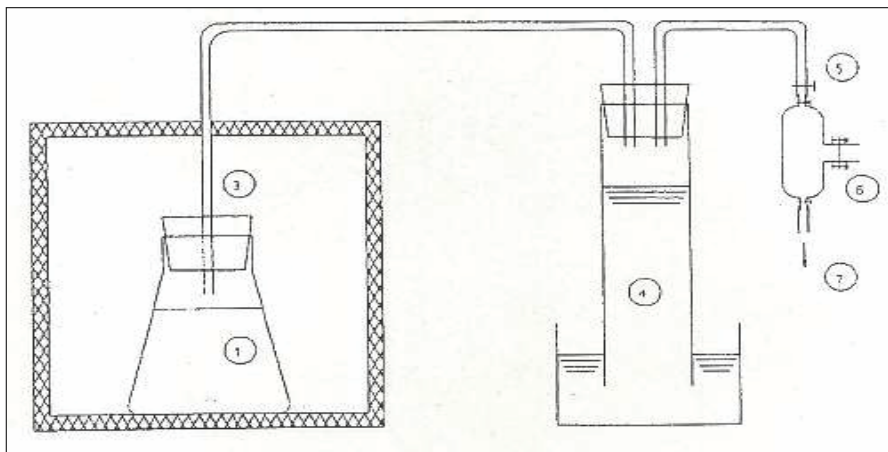


Figure-4: Digester setup

1. Digester
2. Incubator
3. Gas outlet
4. Gas collector
5. Valve
6. Gas Sampling
7. Gas Discharge

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RESULT:

The biogas volume in the gas sampling bag was measured (Table-2& 3). Presence of gas in the gas collector of Positive control indicated that the inoculum was viable and gas displacement was observed both in Positive control and Test.

ASTM D 5511 states that for the test to be considered valid, the positive control must achieve 70 % biodegradation within 30 days. Positive control showed 74.8% biodegradation on the 28th day thus the test is considered valid.

The gas displacement observed after 52 days is as shown in the table below.

Table-2: Biogas volume of the evolved gas during the biodegradation process at 52 days

Samples	Biodegradation Test	Total Volume 52 days (mL)
Paper cup samples (with biodegradable additive)	Inoculum	733
	Positive control	7170
	Paper cup samples	7150



Figure 5: Image of sample before & After Study

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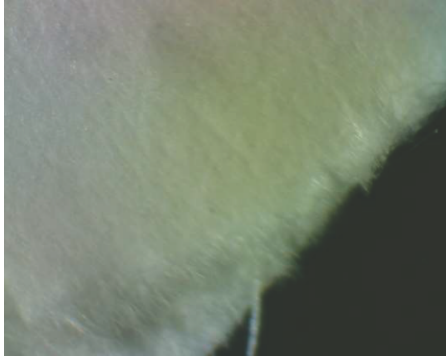
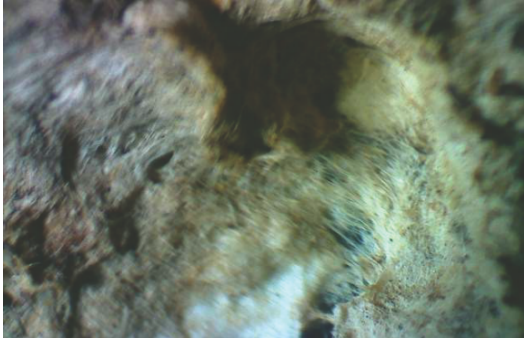




Paper cup (Before)	Paper cup (After)
	
	
	

Figure 6 : Microscopic image of Paper cup samples and After 52 days Incubation Condition

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Table-3: Showing percentage biodegradability of Paper cup samples with respect to positive control Cellulose

Group	Inoculum control	Positive control	PP cups samples
Weight	1000 ml	10.0006 g	10.0250 g
Total volume (ml)	733	7170	7150
% CH ₄	2.00	60.00	62.00
Volume of CH ₄ (ml)	14.66	4302.00	4433.00
weight of CH ₄ (g)	0.01	2.82	2.91
% CO ₂	7.00	38.00	40.00
Volume of CO ₂ (ml)	51.31	2724.60	2860.00
Weight of CO ₂ (g)	0.10	5.39	5.66
Total weight of carbon in grams	0.03	3.57	3.71
Theoretical weight of carbon in grams (Ci)	0.00	3.76	4.46
Biodegradation	-	0.94	0.82
% Biodegradation	-	94.09	82.41

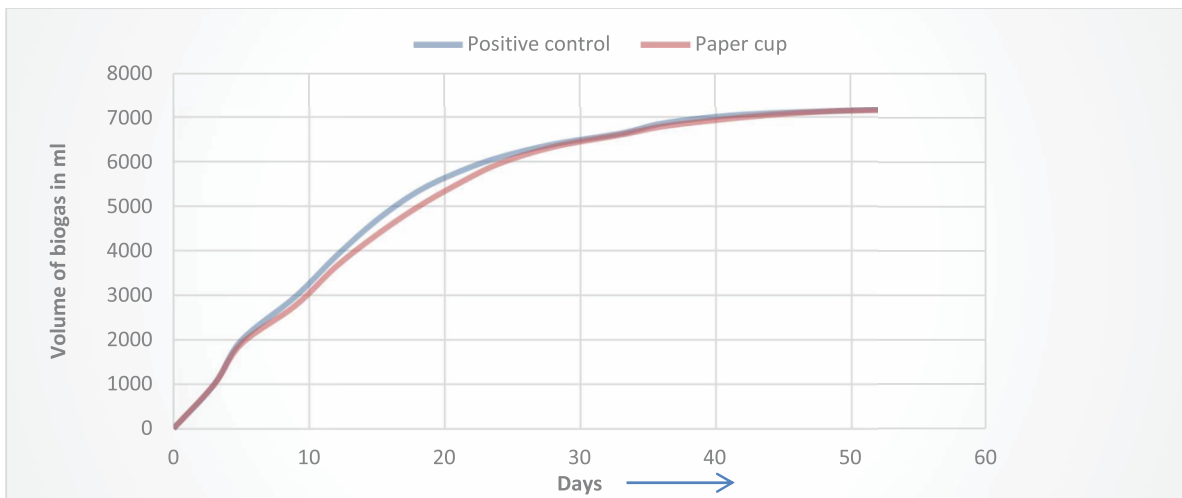
Table 4: Percent weight loss of sample after incubation

Average Initial Weight	10.0250 g
Average Final Weight	0.8141 g
Percent Weight Loss	91.87 %

The above table 3 shows the percent degradation of cellulose (Positive control), Paper cup samples over 52 days of testing. The percent degradation was calculated by the measured cumulative carbon dioxide and methane production from each sample after subtracting carbon

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dioxide evolution and methane evolution from the blank samples at the end of 65 days of testing. Calculations were made based on total organic carbon of Paper cup samples containing (44.45%), and cellulose (37.6%). Biodegradation of the samples determined based on conversion of carbon from the test material to carbon in the gaseous phase (CH₄ and CO₂) can be observed in Graph 1. Table 4 shows the percent loss in average weight of sample after 52 days corresponding to approximately 92% of weight loss.

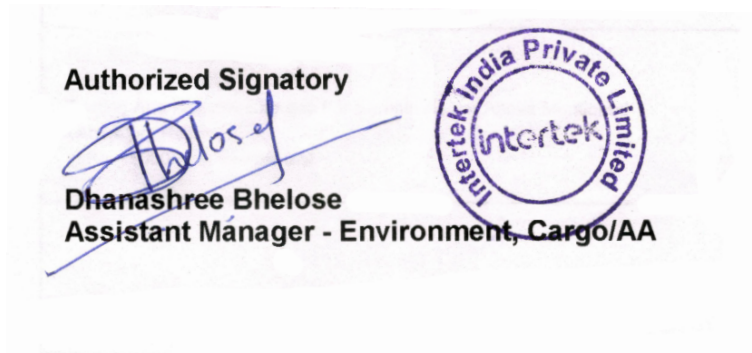


Graph-1: The percent biodegradation of the Paper cup samples determined based on conversion of carbon from the test material to carbon in the gaseous phase (CH₄ and CO₂)

CONCLUSION:

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Considering the cumulative gas production as observed in table 2 and its analysis indicates that the process of biodegradation has occurred. After 52 days of incubation, the level of biodegradation for the positive control (cellulose) was 94.01%, the Paper cup samples showed 82.41% relative to the positive control. The submitted Paper cup samples by Leetha Industries tested under the ASTM D5511-11 method shows biodegradation.



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