

FINAL REPORT

ASTM D5511 Standard Test Method for Determining Anaerobic Biodegradation of Plastic Materials Under High-Solids Anaerobic-Digestion Conditions

ENVIRONMENTAL DIVISION LABORATORY, MUMBAI

INTERTEK INDIA PRIVATE LIMITED

IPL/17025/ENV/QF/7.8/01-03	Issue No.: 01	Amend No.: 00
	Issue Date.: 16.12.2019	Amend Date.: 00.00.0000

Client: LUYI COUNTY JIAMEI COSMETIC CO. LTD.
(Address: Nongfei North Road, Luyi Industrial Park, Luyi county, Henan Province 477200, China)

Sample Registration Date: 21/08/2020

Analysis starting date: 21/08/2020 (pre-conditioning) **Analysis completed on:** 27/10/2020

Name of product: False eyelashes & brush (0.07*35 mm)

Quantity received and packing: 500gms packing

Sample details: PBT with 0.5% of BDP– False eyelashes, makeup brushes and shaving brush (0.07*35mm) sample

Test Required: ASTM D5511 Standard Test Method for Determining Anaerobic Biodegradation of Plastic Materials Under High-Solids Anaerobic-Digestion Conditions

Sampling done by: Sample not drawn by Intertek

Report No. MUM/001478/2020

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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 0.5% BDP – False eyelashes, makeup brushes and shaving brush (0.07*35mm) samples were received on 21/08/2020 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: Test Sample

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PROJECT DESCRIPTION:

PBT 0.5% BDP – False eyelashes, makeup brushes and shaving brush (0.07*35mm) samples were submitted by LUYI COUNTY JIAMEI COSMETIC CO. LTD. Plastic 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 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 allowing 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

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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.

METHODOLOGY:

Inoculum Medium: Remove enough inoculum (approximately 15 kg) from the post-fermentation vessel and mix carefully and consistently by hand in order to obtain a homogeneous medium. Test three replicates each of a blank (inoculum only), Positive control (Reference material) (thin-layer chromatography cellulose), negative control (optional), and the test substance being evaluated.

Manually mix 1000 g wet weight (at least 20 % dry solids) of inoculum in a small container for a period of 2 to 3 min with 15 to 100 g of volatile solids of the test substance or the controls for each replicate. For the three blanks containing inoculum only, manually mix 1000 g of the same inoculum in a small container for a period of 2 to 3 min with the same intensity as was done for the other vessels containing test substance or controls. Determine the weight of the inoculum and test substance added to each individual Erlenmeyer flask accurately. Add the mixtures to a 2-L wide-mouth Erlenmeyer flask and gently spread and compact the material evenly in the flask to a uniform density.

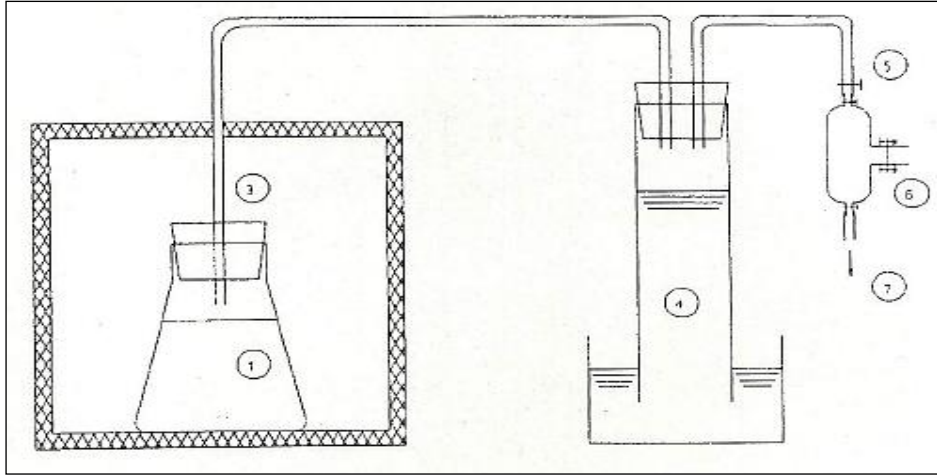
After placing the Erlenmeyer flask in incubator, connect it with the gas collection device. Incubate the Erlenmeyer flasks in the dark or in diffused light at 52°C (62°C) for thermophilic conditions, The incubation time shall be run until no net gas production is noted for at least five days from both the Positive control (Reference material) and test substance reactors. Control the pH of the water used to measure biogas production to less than two by adding HCl.

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ANAEROBIC DIGESTER SETUP FOR THE PLASTIC BIODEGRADATION

The biodegradation testing of sample was performed in the digester as shown in the (Figure-3).



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

Figure-3: Digester setup

RESULT:

The most important biochemical characteristics of the inoculum such as pH, Volatile Fatty Acids, NH₄⁺-N— and dry solids were studied.

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

Parameters	Requirement	Actual results
pH	7.5 to 8.5	7.64
Kjeldahl nitrogen	0.5 to 2 g/kg wet weight	1.40
Dry Solids at 105 °C	>20%	40.00
Volatile Solids at 550 °C	Below 1 g/kg wet weight	0.70

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

ASTM D 5511 states that for the test to be considered valid, the Positive control (Reference material) must achieve 70 % within 30 days with deviation less than 20% of the mean between the replicates.

Positive control (Reference material) showed 71.04% on 28th day with less than 20% of the mean difference between the replicates.

The gas displacement observed after 45 days is as shown in the table below.

Table-2: Biogas volume of the evolved gas during the biodegradation process at 45 days

Biodegradation Test	Total Volume 45 days (mL)
Inoculum	1850
Positive control (Reference material)	9690
PBT with 0.5% of BDP– False eyelashes, makeup brushes and shaving brush (0.07*35mm) sample	4620

Colonization of bacteria at some places were observed under the microscope (Fig-4). This shows the process of biodegradation has begun.

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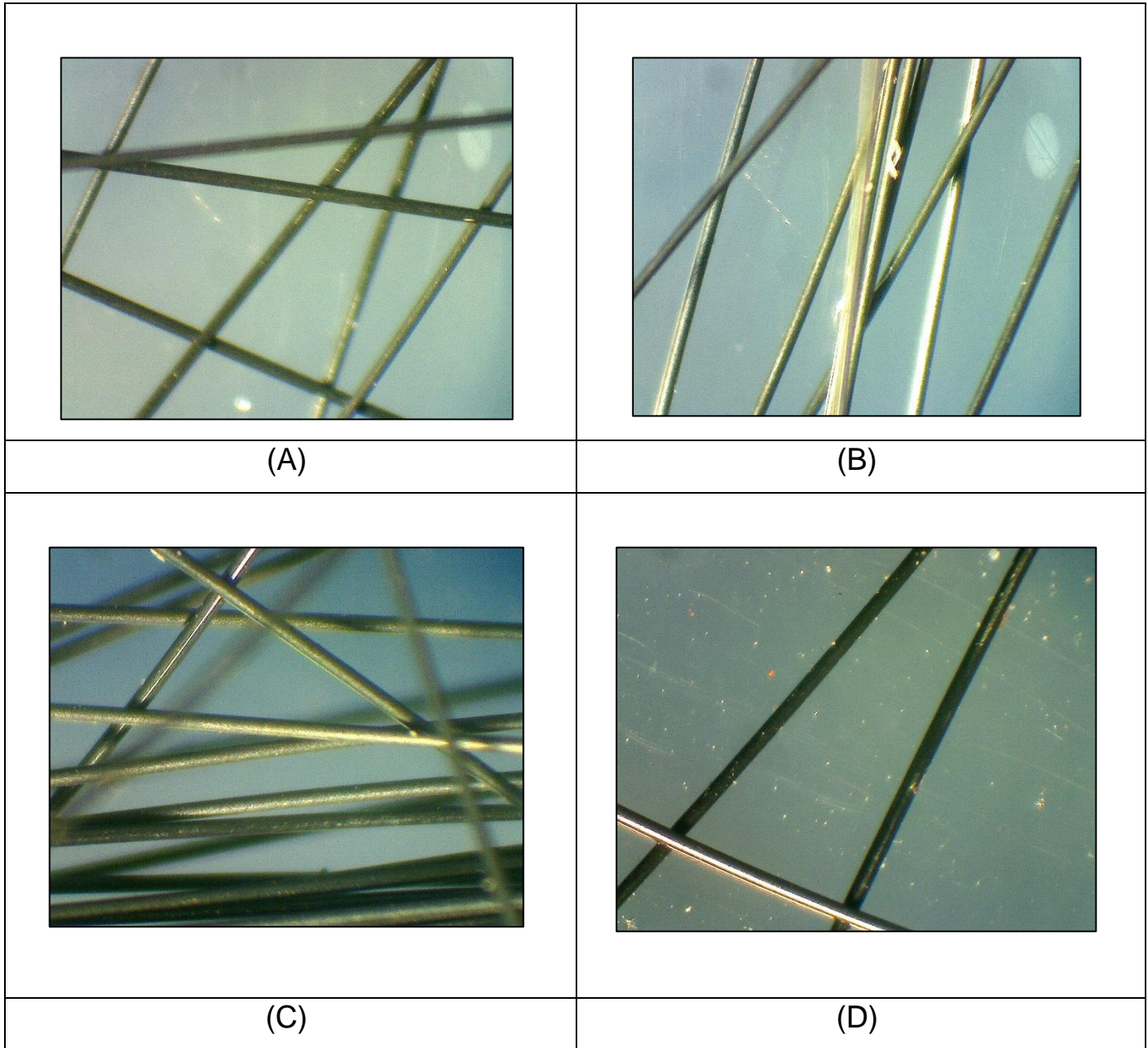


Figure 4: Microscopic image of Test samples Before and After 45 days Incubation Condition

A & B – Unexposed PBT with PBT with 0.5% of BDP– False eyelashes, makeup brushes and shaving brush (0.07*35mm) sample to anaerobic biodegradation process

C & D – Exposed PBT with PBT with 0.5% of BDP– False eyelashes, makeup brushes and shaving brush (0.07*35mm) sample to anaerobic biodegradation process

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The percent biodegradation of Positive control (Reference material) and Test sample was calculated by the measured cumulative carbon dioxide and methane production from each flask after subtracting carbon dioxide evolution and methane evolution from the blank samples at the end of 45 days of testing. Calculations were based on Total Organic Carbon obtained of both Positive control (Reference material) and Test sample.

Table-3: Percentage biodegradability of Test sample with respect to Positive control (Reference material) Cellulose.

Group	Inoculum control	Positive control (Reference material)	PBT with 0.5% of BDP- False eyelashes, makeup brushes and shaving brush (0.07*35mm) sample
Weight	1000 ml	10.0122 g	10.2022 g
Total volume (ml)	1850.00	9690.00	4620.00
% CH ₄	15.20	44.40	18.60
Volume of CH ₄ (ml)	281.20	4302.36	859.32
weight of CH ₄ (g)	0.1845	2.8223	0.5637
% CO ₂	13.00	41.50	14.10
Volume of CO ₂ (ml)	240.50	4021.35	651.42
Weight of CO ₂ (g)	0.4762	7.9623	1.2898
Total weight of carbon in grams	0.2669	4.2666	0.7710
Theoretical weight of carbon in grams (Ci)	-	4.2100	7.0619
Biodegradation	-	0.9500	0.0714
% Biodegradation	-	95.00	7.14

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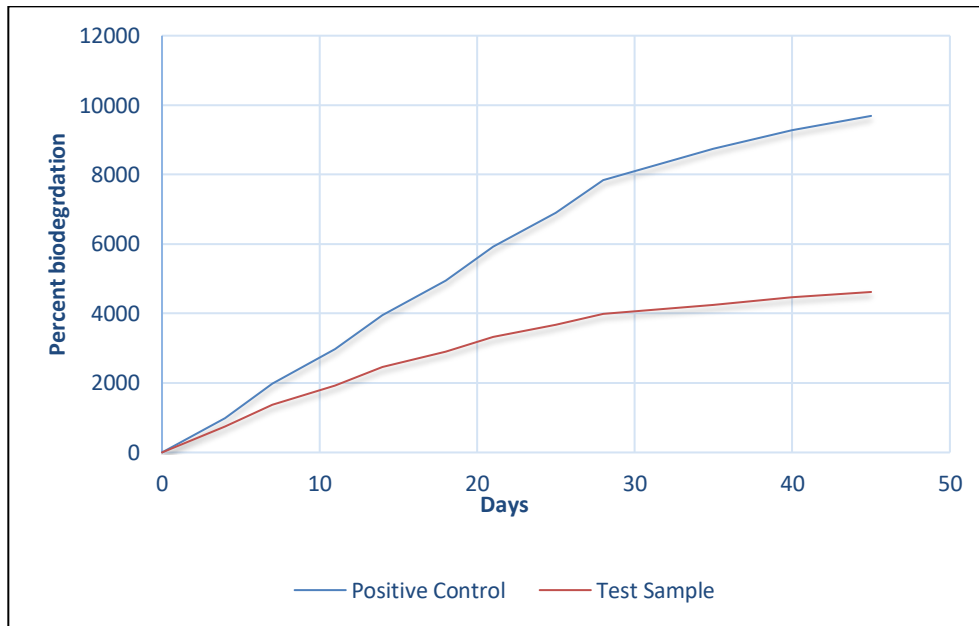
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Table 4: Percent weight loss of PBT with 0.5% of BDP– False eyelashes, makeup brushes and shaving brush (0.07*35mm) sample.

Average Initial Weight (grams)	10.2022
Average Final Weight (grams)	10.0477
Percent Weight Loss (%)	1.51

The Percent weight loss was calculated based on the initial weight and final weight of the test sample after the 45 days study

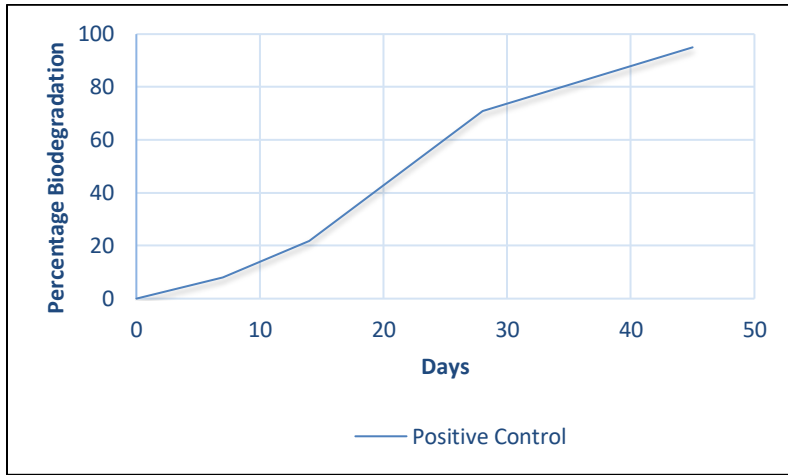
Biodegradation of the samples determined based on conversion of carbon from the test material to carbon in the gaseous phase (CH4 and CO2) can be observed in graph 1 and graph 2a & 2b.



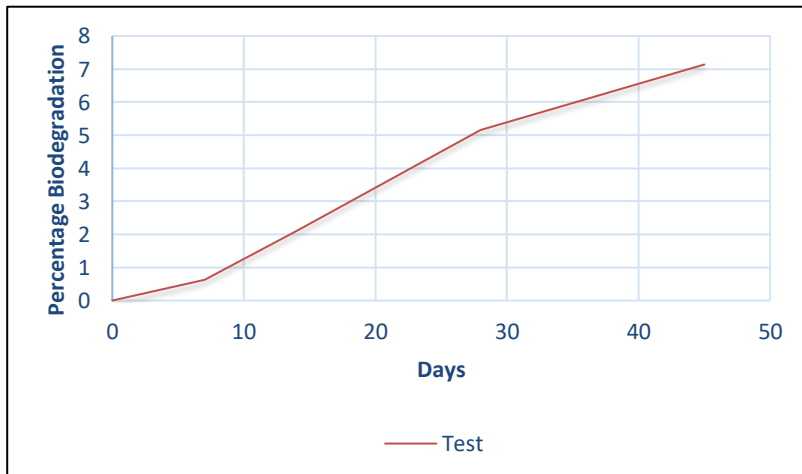
Graph-1: Plot showing Net Biogas Production from Test sample (PBT with 0.5% of BDP– False eyelashes, makeup brushes and shaving brush (0.07*35mm) sample) and Positive control (Reference material- Cellulose)

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Graph-2a: The percent biodegradation of the Positive control (Reference material- Cellulose) determined based on conversion of carbon from cellulose to carbon in the gaseous phase (CH₄ and CO₂)



Graph-2b: The percent biodegradation of the Test sample (PBT with 0.5% of BDP– False eyelashes, makeup brushes and shaving brush (0.07*35mm) sample) determined based on conversion of carbon from the Test material to carbon in the gaseous phase (CH₄ and CO₂)

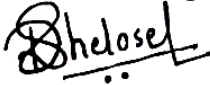
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CONCLUSION:

Considering the cumulative gas production as observed in Table 2 & 3 and its analysis indicates that the process of biodegradation has occurred in **PBT with 0.5% of BDP– False eyelashes, makeup brushes and shaving brush (0.07*35mm) sample**. After 45 days of incubation, the level of biodegradation for the Positive control (Reference material) was **95 %** while the **PBT with 0.5% of BDP– False eyelashes, makeup brushes and shaving brush (0.07*35mm) sample** submitted by **LUYI COUNTY JIAMEI COSMETIC CO. LTD. Plastic** showed **7.14 %**.

Authorized Signatory




Dhanashree Bhelose

Asst. Manager – Business Development & Laboratory Caleb Brett

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