

REPORT NUMBER:



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TEST REPORT

Date: Apr 21, 2020

Sample Description As Declared :

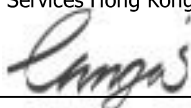
Sample Description : Sample A - 0.38mm PET Film with EP02 1% Biodegradable Additive
Sample B - 0.38mm PET Film with EP02 0.5% Biodegradable Additive

Fibre Content : PE

Date Received/Date Test Started : JAN 24, 2020

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For and on behalf of
Intertek Testing Services Hong Kong Limited



Teddy Y. N. Chung
Director

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Tests Conducted (As Requested By The Applicant)

- 1 Standard Test Method For Determining Anaerobic Biodegradation Of Plastic Materials Under High-Solids Anaerobic-Digestion Conditions (ASTM D5511-18):

PROJECT DESCRIPTION:

Sample A - 0.38mm PET Film with EP02 1% Biodegradable additive and Sample B - 0.38mm PET Film with EP02 0.5% Biodegradable additive samples were submitted 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 1) mixed with household waste. 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 1: 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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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.

ANAEROBIC DIGESTER SETUP FOR THE PLASTIC BIODEGRADATION

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

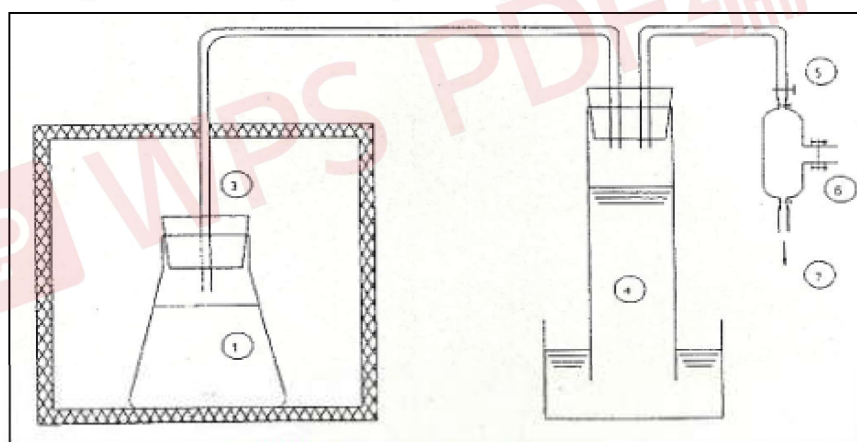


Figure-2: Digester setup

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

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RESULT:

The most important biochemical characteristics of the inoculum such as pH, Volatile Fatty Acids, NH_4^+-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	7.92
Kjeldahl nitrogen	0.5 to 2 g/kg wet weight	1.54
Dry Solids at 105 °C	>20%	39.0
Volatile Solids at 550 °C	Below 1 g/kg wet weight	0.70

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 D5511 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 70.73% on 24th 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.

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

Biodegradation Test	Total Volume 45 days (mL)
Inoculum	1750
Positive control (Reference material)	9575
Sample A - 0.38mm PET Film with EP02 1% Biodegradable additive	4870
Sample B - 0.38mm PET Film with EP02 0.5% Biodegradable additive	4520

Colonization of bacteria at some places were observed under the microscope (Fig-3 & 4). This shows the process of biodegradation has begun.

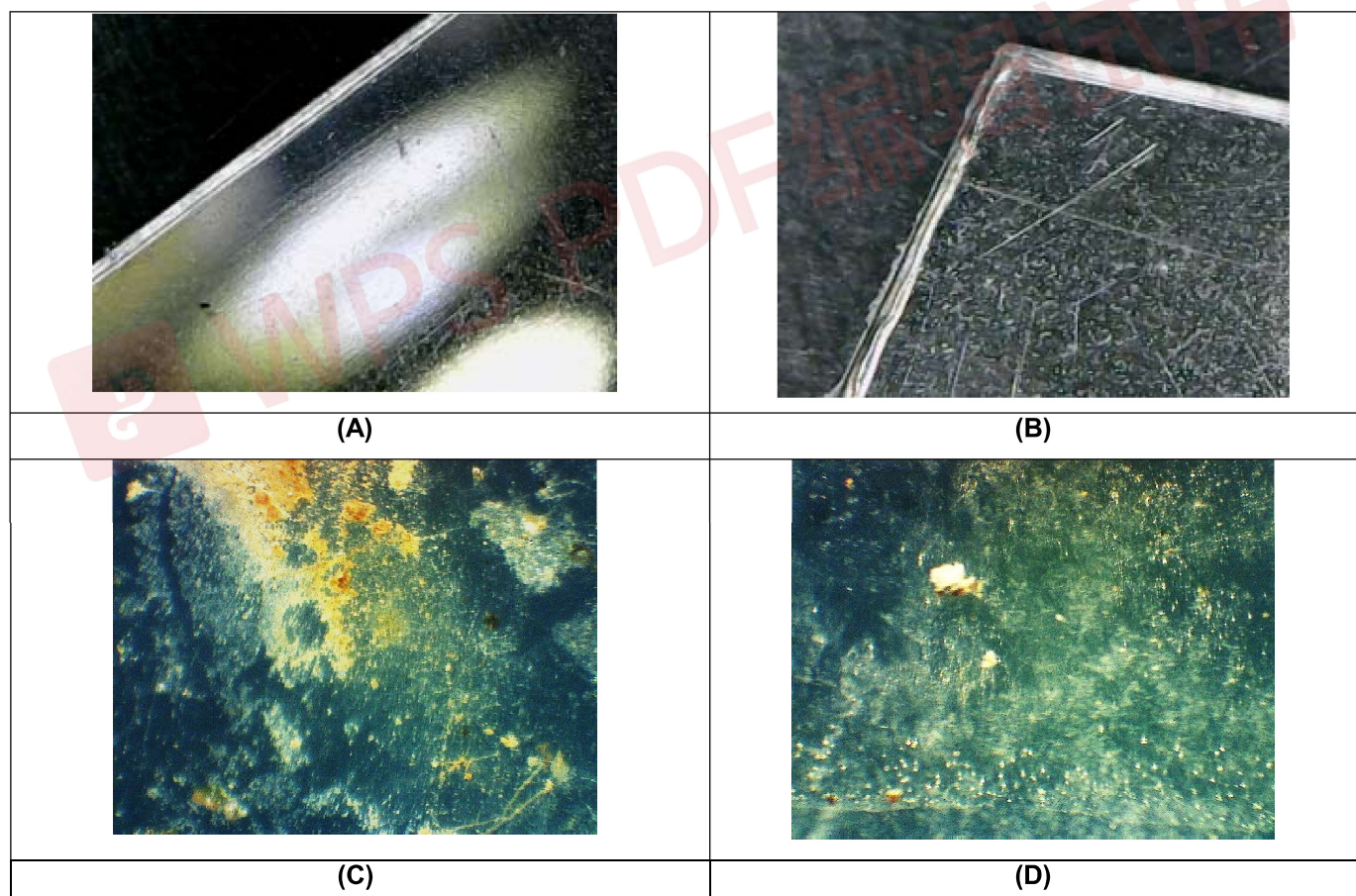


Figure 3: Microscopic image of Test samples Before and After 45 days Incubation Condition
A & B – Unexposed Test Sample A to anaerobic biodegradation process
C & D – Exposed Test Sample A to anaerobic biodegradation process

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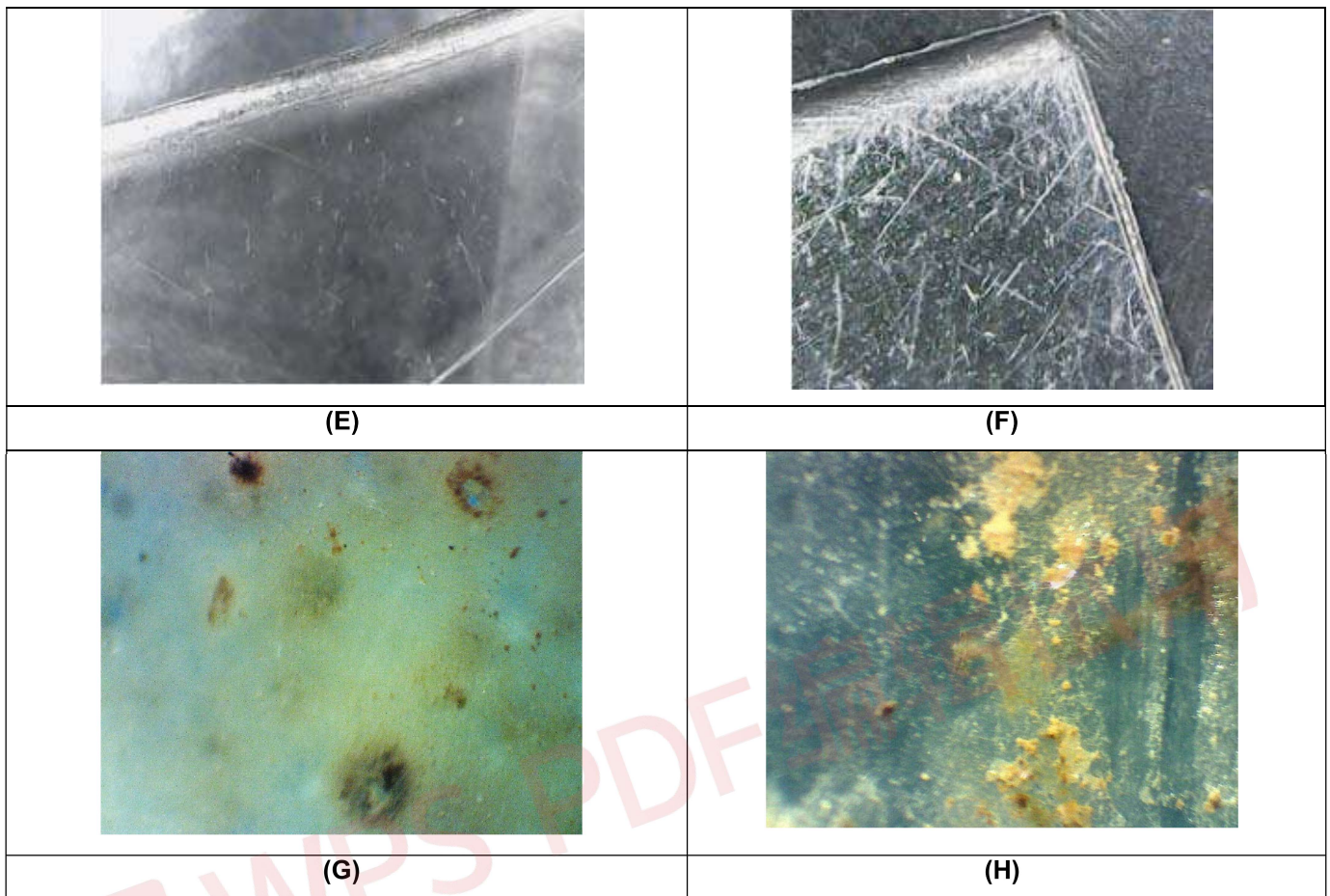


Figure 4: Microscopic image of Test samples Before and After 45 days Incubation Condition
E & F– Unexposed Test Sample B to anaerobic biodegradation process
G & H – Exposed Test Sample B to anaerobic biodegradation process

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.

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

Group	Inoculum control	Positive control (Reference material)	Sample A	Sample B
Weight	1000 ml	10.0788 g	9.9330 g	9.7828 g
Total volume (ml)	1750.00	9575.00	4870.00	4520.00
% CH ₄	12.90	43.10	16.00	15.20
Volume of CH ₄ (ml)	225.75	4126.83	779.20	687.04
weight of CH ₄ (g)	0.1481	2.7072	0.5112	0.4507
% CO ₂	9.40	40.20	13.00	12.70
Volume of CO ₂ (ml)	164.50	3849.15	633.10	574.04
Weight of CO ₂ (g)	0.3257	7.6213	1.2535	1.1366
Total weight of carbon in grams	0.1990	4.0882	0.7218	0.6449
Theoretical weight of carbon in grams (Ci)	-	4.24	6.43	6.42
Biodegradation	-	0.9173	0.0813	0.0695
% Biodegradation	-	91.73	8.13	6.95

Table 4: Percent weight loss of Sample A - 0.38mm PET Film with EP02 1% Biodegradable additive

Average Initial Weight (grams)	9.9330
Average Final Weight (grams)	9.7778
Percent Weight Loss (%)	1.56

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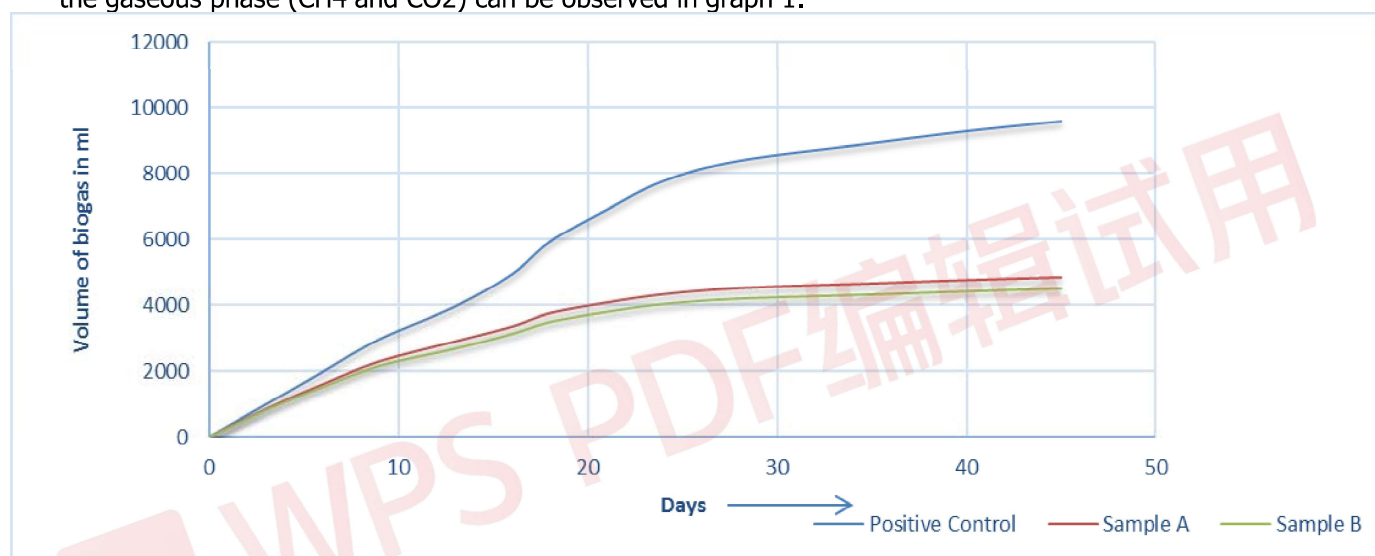
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Table 5: Percent weight loss of Sample B - 0.38mm PET Film with EP02 0.5% Biodegradable additive

Average Initial Weight (grams)	9.7828
Average Final Weight (grams)	9.6556
Percent Weight Loss (%)	1.30

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.



Graph-1: The percent biodegradation of the Test 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 Sample A - 0.38mm PET Film with EP02 1% Biodegradable additive and Sample B - 0.38mm PET Film with EP02 0.5% Biodegradable additive. After 45 days of incubation, the level of biodegradation for the Positive control (Reference material) was 91.73 % while the Sample A and Sample B showed 8.13% and 6.95 % respectively.

Remark: The test was subcontracted to laboratory under Intertek group.

End of Report

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