STUDY ON CARBON BLACK DISPERSION GRADE IN POLYETHYLENE GEOMEMBRANE

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ABSTRACT

Two methods are used to prepare polyethylene geomembrane samples. One is microtome method and the other is melt compression method. The dispersion grade of these samples are determined by Lynx Stereo microscopy and the results are compared. The study shows that there are some differences when using different sample preparation methods and it can help to get more reliable results when using microtome method.

Keywords: Polyethylene geomembranes, carbon black dispersion, dispersion grade, microtome method, melt compression method

INTRODUCTION

Geomembrane is a kind of polymer membrane materials widely used in the engineering field owing to its extremely low water penetration (10^{-13}~10^{-14} m/s). Compared with traditional waterproof materials, geomembrane possesses extra advantages such as high strength, reliable joints, good flexibility and deformation adaptation.

Among series of geomembranes, polyethylene (PE) geomembranes especially MDPE/HDPE geomembranes are with better comprehensive performance, such as excellent water resistance, longer elongation, higher strength, better flexibility at low temperature, much lighter, better welding properties and so on. In this way, PE geomembranes could be widely used in many important construction projects, for example, municipal sanitary landfills, heap leaching of gold ores and treatment of tailings in mine, waterproof in chemical works and petrochemical industry, high-speed railway and so on.

The aging-resistance property is deeply concerned for the MDPE/HDPE geomembranes is usually decomposed in chemical circumstance and direct sunlight. The common way to protect geomembranes from UV degradation is to add carbon black (CB) into the products. In addition, CB should be added to a certain level (usually 2.0~3.0%) and dispersed uniformly, as a result, it is necessary to determine the dispersion of CB in the PE geomembranes. At present, ASTM D 5596-03 , ISO 18553-2002 and GB/T18251-2000[1-3] are used to determine the dispersion of CB in polyolefin geosynthetics. In this paper, both microtome and melt compression method are used to prepare polyethylene goemembrane samples. Then, the dispersion of CB in the PE geomembranes is determined according to ASTM D 5596-03, problems occurred in the process were discussed at the same time.

EXPERIMENT

Materials

The geomembranes used in this paper are HDPE smooth geomembranes with a thickness of 1.5mm.

Instruments

Instruments used in this paper are listed as follows:
Lynx Stereo dynascope, Vision Engineering Ltd;
Miniview image analysis system, Microsoft Corporation；
Ultra-microtome, MT-6000, Du Pont Company;
High and low temperature oven, Guangzhou Espec Environmental Instrument Co., Ltd.

Experiment Process

Sample preparation

The preparation method of sample has effects on CB dispersion. Microtome method and melt compression method are generally used. ASTM D 5596-03 standard adopts microtome method while ISO 18553-2002 and GB/T 18251-2000 involve both two methods but using melt compression as the arbitration method. In this paper, microtome method and melt compression method are both employed to

analyze the effect of preparation on the determination of the dispersion of CB.

For melt compression, ten samples with area of $2mm^2$ were cut as thin as possible from different part of the product. The samples were arranged on two slides at same intervals separately and covered with another two slides. Each group of slides was clipped by a spring and held in an oven at 200℃ for 1h. Then the slides were removed from the oven and cooled naturally. The thickness of the films prepared by melt compression should fall between 8μm to 20μm.

For microtome method, a 4mm×2mm×2mm specimen was cut from the product and stuck to the holder of the ultra-microtome. The specimen and the knife of the ultra-microtome were cooled for 15min with liquid nitrogen. Ten ultra section with thickness of 8μm~20μm were then cut from the specimen and arranged on two slides at same intervals separately. Cover the two slides with another two slides.

Determination of the CB dispersion

Microscopy observations of the CB particles and clusters were performed at a magnification of 100 with transmission light. The images of maximum CB particles and clusters were saved and analyzed with miniview image analysis system. These particles and clusters were then classified by comparing their area with the ASTM D 5596-03 and the spectrum in D35.

RESULTS AND DISCUSSIONS

Morphology of CB Particles and Clusters in Prepared Samples

Series of experiments and observations we have carried out indicate that the preparation methods produce different effects on the experiment results. Here, one sample prepared by each method was investigated typically.

Morphology of CB dispersion in sample prepared with melt compression method is shown in Figs. 1(×15) and 2(×100). Fig. 3(×15) and Fig. 4(×100) are morphology of CB in sample prepared by ultrathin sectioning.

Fig. 1 Morphological image of carbon black particles and carbon black agglomerates in specimen 4# made by compressing

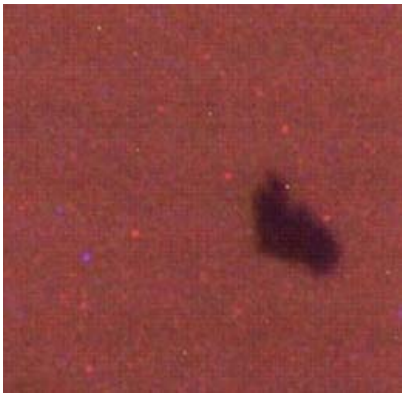

Fig. 2 Morphological image of the biggest carbon black particle and carbon black agglomerate in specimen 4# made by compressing

Fig. 3 Morphological image of carbon black particles and carbon black agglomerates in specimen 3# made by microtome

Fig. 4 Morphological image of the biggest carbon black particle and carbon black agglomerate in specimen 3# made by microtome

As can be seen, the sample prepared by melt compression shows a smoother surface than that of the sample prepared by ultra-sectioning. This is originated from that the slice produced by the microtome is so thin that it could crinkle easily. A problem worthy to be pointed out is that when prepared samples by melt compression, a space should be kept between two slides to deflate, otherwise, amount of bubbles would form at the surface of samples.

Dispersion of CB in samples prepared by melt compression

Classification of CB particles and clusters in samples prepared by melt compression is listed in Table 1.

Based on the CB particles and clusters classifications of the ten samples, this product is assessed as grade 2~3.

Table 1 Test data of the samples made by melt compressing

Sample	Area of maximum CB particles and clusters（μm²）	Classification
1	5958	3
2	9025	3
3	4629	3
4	4656	3
5	1588	2
6	8924	3
7	2325	2
8	1554	2
9	2884	2
10	6582	3

Dispersion of CB in samples prepared by microtome

Classification of CB particles and clusters in samples prepared by microtome is listed in Table 2.

Table 2 Test data of the samples made by microtome

Sample	Area of maximum CB particles and clusters（μm²）	Classification
1	1554	2
2	1895	2
3	1363	2
4	1166	2
5	784	1
6	982	2
7	1653	2
8	1231	2
9	1159	2
10	886	1

Based on the CB particles and clusters classifications of the ten samples, this product is assessed as grade 1~2.

The influence factor of sample preparation method on CB dispersion grade

In normal case, CB particles cannot disperse uniformly but aggregate in the geomembranes. The schematic diagram of a single CB particle is shown in Fig. 5. The diameters of CB particles usually fall between 15 to 60 mm. Fig. 6 is the schematic diagram of aggregated CB clusters which is the common state in the product. CB is one of the most effective and widely used ultraviolet absorbents. The efficiency of CB as an ultraviolet absorbent is decided by the dispersion of the particles in the product, i.e. the size and amount of CB clusters. Meanwhile, stress concentration would appear if the particles aggregated greatly leading to the formation of little cracks and finally the break of the product. As a result, the dispersion of CB particles is a reflection of the aging-resistance properties and mechanical properties.

Fig. 5 Granoplasm of carbon black

Fig. 6 Carbon black agglomerate

As have been shown before, a same product is classified in different grades with different preparation methods. The grades of samples prepared by melt compression are bigger than that prepared by microtome. This is originated from the melt process of the melt compression method. Under pressure, the polyethylene melt could flow away. During this process, CB particles and clusters could redistribute and merge into bigger clusters leading to the increase of dispersion grade (Fig. 7). Furthermore, the area of CB particles and clusters of the melt could enlarge under pressure which could also increase the dispersion grade (Fig. 8). However, for the sample prepared by microtome, the structure of the sample is frozen by liquid nitrogen, the size and distribution of CB particles and clusters remain unchanged. In this way, the CB dispersion grade could be determined more precisely.

Fig. 7 The free sketch of carbon black particle aggregation

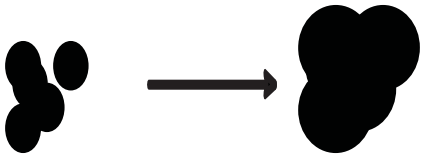

Fig. 8 The free sketch of carbon black particle and carbon black agglomerate squashed

CONCLUSIONS

1) The dispersion of CB particles and clusters in geomembranes is investigated using Lynx Stereo dynascope microscopy and miniview image analysis system. The size of CB particles and clusters is obtained simultaneously.
2) With the sample prepared by microtome, the dispersion of CB remains unchanged. On the other hand, melt compression process would change the distribution of CB particles and clusters, leading to the increase of the dispersion grade. The dispersion grade could be determined more precisely with the sample prepared by microtome.
3) It is suggested to prepared sample by microtome. Considering the high price and the complexity of the microtome, labs without microtome could prepare samples by melt compression instead.
4) The preparation method of the sample should be specified in the report to make the results more comparative.

REFERENCES

ASTM D 5596-03， Standard Test Method for Microscopic Evaluation of the Dispersion of Carbon Black in Polyolefin Geosynthetics.

ISO18553-2002，1 Method for the Assessment of the Degree of Pigment or Carbon Black Dispersion in Polyolefin Pipes, Fittings and Compounds.

GB/T18251-2000 Method for the Assessment of Pigment or Carbon Black Dispersion in Polyolefin Pipes, Fittings and Compounds.