

# AN INVESTIGATION INTO THE PHYSICAL PROPERTIES OF POLYETHYLENE GAS PIPES BY THE INCORPORATION OF ANTI- RODENT MASTERBATCHES

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## ABSTRACT

The incorporation of several anti-rodent masterbatches into polyethylene (PE) granules used for gas pipes and the effects of the masterbatches on the physical properties of the pipes were studied. Four commercial anti-rodent masterbatches from different international companies including, Clariant, Alchemix, PolyOne, and Newgen were used in this research. Each of the masterbatch was primarily dry-blended with PE granules by a laboratory-scale tumbling mixer. The concentration of anti-rodent masterbatches used in the PE blends was fixed according to the recipe recommended by the producers as 1, 2.5, 3, and 4 phr for the masterbatches of Clariant, Alchemix, PolyOne, and Newgen respectively. PE granules and masterbatch blends were separately fed into the hopper of a pipe extruder and a length of a hundred and fifty meters of the pipe with the outside diameter (O.D.) of 25 millimeters was continuously produced for each anti-rodent-added PE compound. Two groups of experiments were planned for this investigation; laboratory experiments including impact resistance, tensile elongation, environmental stress cracking (ESCR), melt flow rate (MFR), and differential scanning calorimetry (DSC) were carried out on the control (anti-rodent free) and anti-rodent-added PE100 test samples prepared in the laboratory. Also, a group of factory tests such as burst, hydrostatic (100, 165, and 1000 hr), slow crack growth (SCG), and oxygen induction time (OIT) were performed on the control and anti-rodent-added pipe samples.

**Keywords:** Anti-rodent Materials, Polyethylene Pipes, Rodent Attacks, Melt Blending

## INTRODUCTION

Occasionally, there have been reports of rodent attacks on a number of domestic facilities such as plastic covers of wires and cables, agricultural polymer films, and plastic pipes with O.D. equal and less than 25 mm in some rural and urban areas. The reality is that the rodents' front teeth are continuously growing and these animals are

forced to trim them by gnawing any suitable materials they found around such as wooden objects and then by gnawing any suitable materials they found around such as wooden objects and plastics. From the economics and safety viewpoints, it is not wise to wait and see the plastic objects fail, and then we just replace them with new ones. It is practiced that the incorporation of a kind of additive known as

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anti-rodent masterbatch in some polymeric compounds such as PVC and PE could effectively protect them from rodent attacks [1-3].

According to a research conducted by RIPI, it has been found that rat attacks are the primary cause of gas leakage from PE pipes in some locations of Iran's gas distributing system. A thorough literature survey shows that one of the solutions that could effectively prevent the attacks is the melt blending of an anti-rodent material with PE granule before pipe extrusion [1-3]. However, Iran's polyethylene gas pipe standard, because of the importance of the pipe safety, does not allow the addition of any chemicals to polyethylene pipes.

Historically, anti-rodent masterbatches have been used in the composition of raw materials for manufacturing wire and cable covers, agricultural polymer films, and irrigation plastic pipes but not in polyethylene gas pipes. It has been experienced that the mentioned masterbatches are able to strongly reduce the frequency of rodent attacks on plastic materials [1-3]. Commonly, anti-rodent-added plastic objects are not required to have high level of physical and mechanical properties; however, polyethylene gas pipes are obliged to pass a series of standard strength-measuring tests such as burst, hydrostatic, impact, and so on. In the present study, the effect of four commercial anti-rodent masterbatches on the strength and physical properties of PE gas pipes are examined. A series of laboratory experiments plus several factory tests have been performed on anti-rodent-added and pure polyethylene compounds.

## EXPERIMENTAL

### Materials

Black polyethylene granules of PE100 produced by Borouge Company in the United Arab Emirates (UAE) with a MFR of 0.23 g/10min (at

190 °C, 5kg) and a density of 0.958 g/cm<sup>3</sup> were used in this research. Anti-rodent masterbatches manufactured by four different companies including PolyOne (P), Clariant (C), Alchemix (A), and Newgen (N) were prepared and used in the investigation. All the masterbatches except Newgen's consisted of 95% LDPE and 5% active materials. The Newgen anti-rodent masterbatch was made of 95% polymer blend of ethylene-vinyl acetate copolymer (EVA) and LDPE and 5% active material.

### Sample Preparation and Characterization

Four polyethylene compounds were made on bench scale by melt mixing the black PE100 granules with each of the anti-rodent masterbatches using a laboratory-scale twin screw extruder. The concentration of the anti-rodent masterbatches in polyethylene compounds with the sample codes of 1, 2, 3, and 4 were 4, 3, 1, and 2.5 phr respectively (see Table 1).

**Table1: Abbreviated codes of anti-rodent masterbatches and their recommended concentrations of use in the PE sample**

| Sample code | Abbreviation of anti-rodent manufacturers | Concentration (phr) |
|-------------|-------------------------------------------|---------------------|
| 1           | N                                         | 4                   |
| 2           | P                                         | 3                   |
| 3           | C                                         | 1                   |
| 4           | A                                         | 2.5                 |
| 0           | -                                         | 0                   |

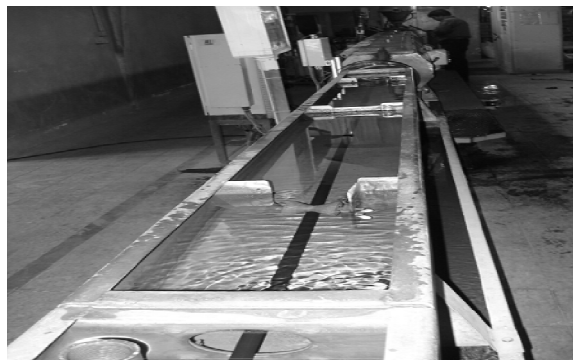
In order to prepare test samples for tensile, impact, ESCR, DSC, and MFR experiments, three PE sheets with the dimensions of 15 cm×15 cm×3 mm were prepared from each anti-rodent/polyethylene compound by using a laboratory hot-presser. The sheets were then cut into rectangular samples with the dimensions of 13 cm×3 cm×3 mm by a suitable cutter. As the tensile test samples were required to be dumbbell shape, each rectangular sample was firmly mounted in a dumbbell-shape

metallic-frame and was trimmed by a cutting-machine into a dumbbell configuration. The impact and ESCR rectangular test samples with the dimensions of 63 mm×12.7 mm and 38 mm×12.9 mm respectively were prepared in the same way.

The tensile test samples were (according to ASTM D638) individually mounted between the fixtures of a tensile tester machine (Zwick, Germany) and were extended at a speed of 50 mm/min to break [4]. The sample tensile strength was automatically calculated by the tensile machine computer program. The impact test samples were (according to ASTM D256) separately mounted in an Izod-impact tester machine (Zwick, Germany) and forced to break with the machine hammer. The impact strength of the samples was read from the gage of the impact tester. In order to perform ESCR experiments on each compound, five rectangular samples were notched with a special notch-making machine (CEAST, Italy) according to ASTM D1693. The notched samples were folded at their notch positions in a U-shaped form and mounted in a clamp to keep them firm. The clamp with the five samples was then immersed in 10% Igepal solution thermostatically controlled at 50 °C. The samples were checked every 24 hours for the growth of their notches. If a notch grew and caused the sample to break apart, its time of breakage would be recorded. The DSC experiment was carried out in the temperature range of 25 °C to 250 °C at a heat rate of 20 °C /min by a DSC instrument (Mettler, USA). MFR tests were also conducted according to ASTM D1238 under the conditions of 190 °C and 5 kg weight by an MFR tester (CEAST, Italy).

PE pipe samples were prepared in Jahad Zamzam factory. A length of about one hundred and fifty meters of polyethylene gas pipes with the O.D. of 25 mm was manufactured by using each anti-rodent/polyethylene compound. PE100 granules and the anti-rodent master-

batches were dry-blended before being fed to the hopper of the pipe extruders. Table 1 shows the concentration of anti-rodent masterbatches used. Sample code 0 refers to the control sample. Figure 1 shows the water-cooling bath of the factory in which a line of anti-rodent-added pipe was cooled.



**Figure 1: The water cooling bath in the pipe factory showing a line of pipe running in.**

After manufacturing the anti-rodent-added PE pipes, their internal and external surfaces were visually examined by axially cutting the pipes. These inspections showed that all the pipes except the one containing Newgen masterbatch had smooth surfaces. The problem detected in the pipe containing Newgen masterbatch was the existence of some bubbles on the internal surface of the pipe. These bubbles were recognized as failure and so this anti-rodent masterbatch and the corresponding compound were withdrawn from the experiments. Thus, the result of burst test of the sample pipe with code 1 was not included in Table 2.

**Table 2: Burst test results**

| Sample code | Burst result (bar) |
|-------------|--------------------|
| 2           | 75                 |
| 3           | 80                 |
| 4           | 74                 |
| "0"         | 76                 |

An investigation was then conducted to find out the reason for bubble formation by Newgen anti-rodent masterbatch. It was revealed that since Newgen Company uses EVA (ethylene-

vinyl acetate copolymer) together with LDPE in the preparation of its anti-rodent masterbatch, and EVA requires a lower processing temperature than PE, so EVA was degraded and produced gas during the pipe extrusion process.

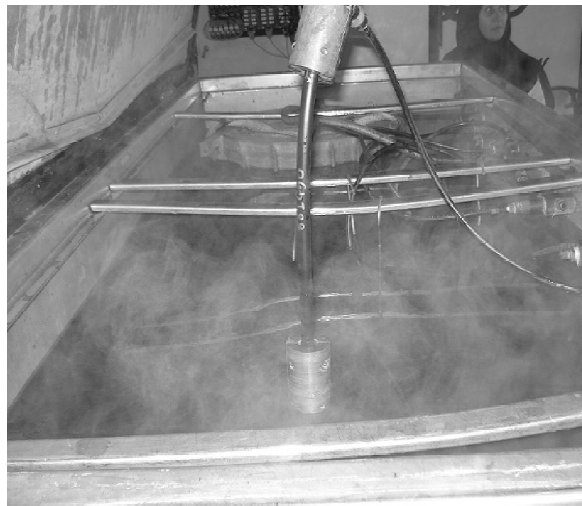
The burst, hydrostatic, OIT, and SCG tests were carried out in the pipe factory [5-7]. The burst test was performed to measure the maximum hydrostatic pressure that the pipe could tolerate at room temperature. To do the test, the pipe samples with a length of 40 cm were prepared by cutting. Both ends of the pipe samples were threaded and a special flange was connected to each end. Then, a high pressure water hose was attached to the pipe and the pressure inside the pipe was increased stepwise by a water pump until the pipe burst and the burst pressure was recorded. Figure 2 shows the pipe arrangements for burst tests and Table 2 presents the burst results. Hydrostatic test is similar to burst test and used to show if a sample pipe can tolerate a predetermined pressure for a specified time period at a high temperature.



**Figure 2: Pipe sample arrangements for burst test**

In order to perform the hydrostatic test, the pipe samples were threaded at both ends, attached to flanges at each end, and connected to a high pressure water hose. Then, the pipes were immersed in a hot water tank held at the temperature of 80 °C. When a pipe burst at a

certain time, its burst time would be recorded. Figure 3 shows the hot water tank and the pipe arrangements for the hydrostatic tests. All the pipe samples successfully passed the hydrostatic tests without being burst.



**Figure 3: Hot water tank arrangement for pipe hydrostatic tests**

The OIT test measures the time period of PE thermal degradation at a specified temperature and can be measured by a DSC instrument. In this research, the OIT tests were carried out by a Mettler DSC at 200 °C. First, a small PE sample (5-10 mg) was heated from room temperature up to 200 °C at the heat rate of 15 °C/min under the purge of nitrogen gas. When the temperature reached 200 °C, the test continued isothermally and at the same time the purge gas was switched to oxygen. After the degradation peak fully appeared, the test was stopped. The time elapsed from the moment of switching the purge gas to oxygen to the time when the degradation peak appeared was recorded as OIT results.

## RESULTS AND DISCUSSION

A series of laboratory experiments including impact resistance, tensile elongation, ESCR, MFR, and DSC were performed on each anti-rodent-added as well as control polyethylene compound (anti-rodent-free PE100). After 1000

hrs of continuous ESCR experiments in 10% Igepal solution at 50 °C no cracks were seen growing among five different groups of the rectangular samples prepared from anti-rodent-added and control PE compounds. The ESCR results showed that the addition of the anti-rodent additive had no adverse effects on the ESCR properties of the compounds.

The results of tensile strength and Izod impact are shown in Tables 3 and 4 respectively. As can be seen, there are not meaningful differences between the tensile strength of the control compound and those of anti-rodent-added PE compounds, revealing that the anti-rodent masterbatches did not significantly change the tensile properties of PE100. Similar results were also found regarding the effect of adding anti-rodent to the compounds on their impact strength.

**Table 3: Tensile strength of anti-rodent-added and pure PE compounds**

| Sample Code | Tensile strength, stress at break (MPa) |
|-------------|-----------------------------------------|
| 2           | 1.03                                    |
| 3           | 1.14                                    |
| 4           | 1.07                                    |
| "0"         | 1.14                                    |

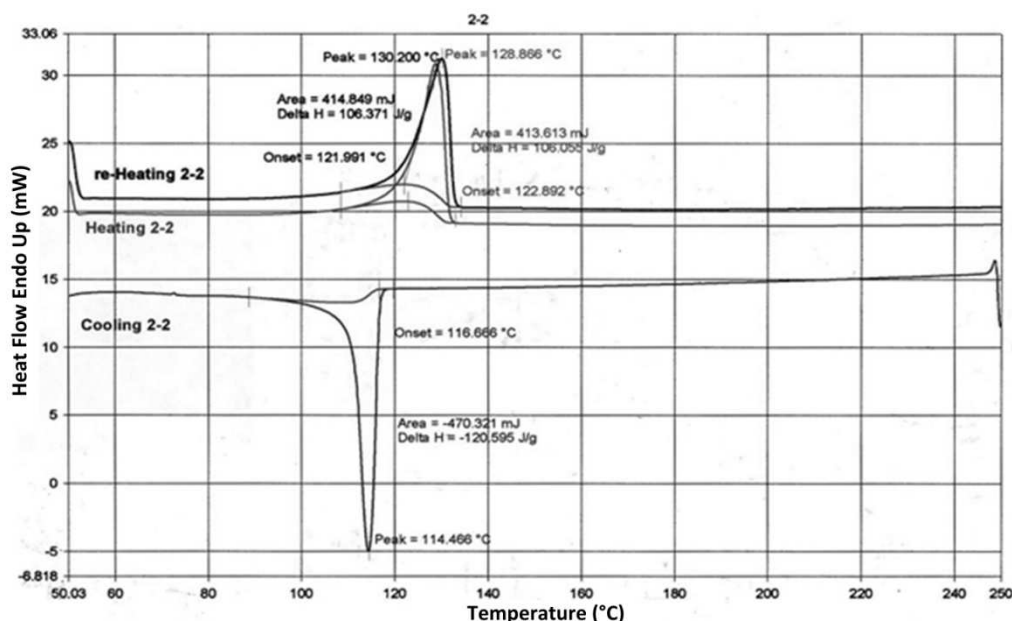
**Table 4: Izod impact strength of anti-rodent-added and pure PE compounds**

| Sample Code | Impact strength, kg.cm/cm |
|-------------|---------------------------|
| 2           | 40.5                      |
| 3           | 40.4                      |
| 4           | 33.5                      |
| "0"         | 38                        |

The DSC analysis carried out on the anti-rodent-added and control compounds (Figure 4-7) showed no shifts in the positions of the melting and crystallization peaks of the compounds. This confirms that the anti-rodent-added and anti-rodent-free poly-ethylene compounds are dominated by the same morphology.

The results of the impact tests showed no meaningful differences between the modified and control compounds. This means no phase segregation occurs in the modified compounds.

A series of standard quality control tests such as burst, hydrostatic of 100, 165, and 1000 hours at 80 °C, SCG, OIT at 2.0 °C, etc. was performed on the pipe samples. Table 2 shows the results of burst test for all the samples except for the sample code 1. Although some small differences may exist among the burst test figures, these differences are marginal and cannot endanger the safety of the gas pipes [8].



**Figure 4: DSC thermogram of the PE100 compound containing the anti-rodent of PolyOne.**

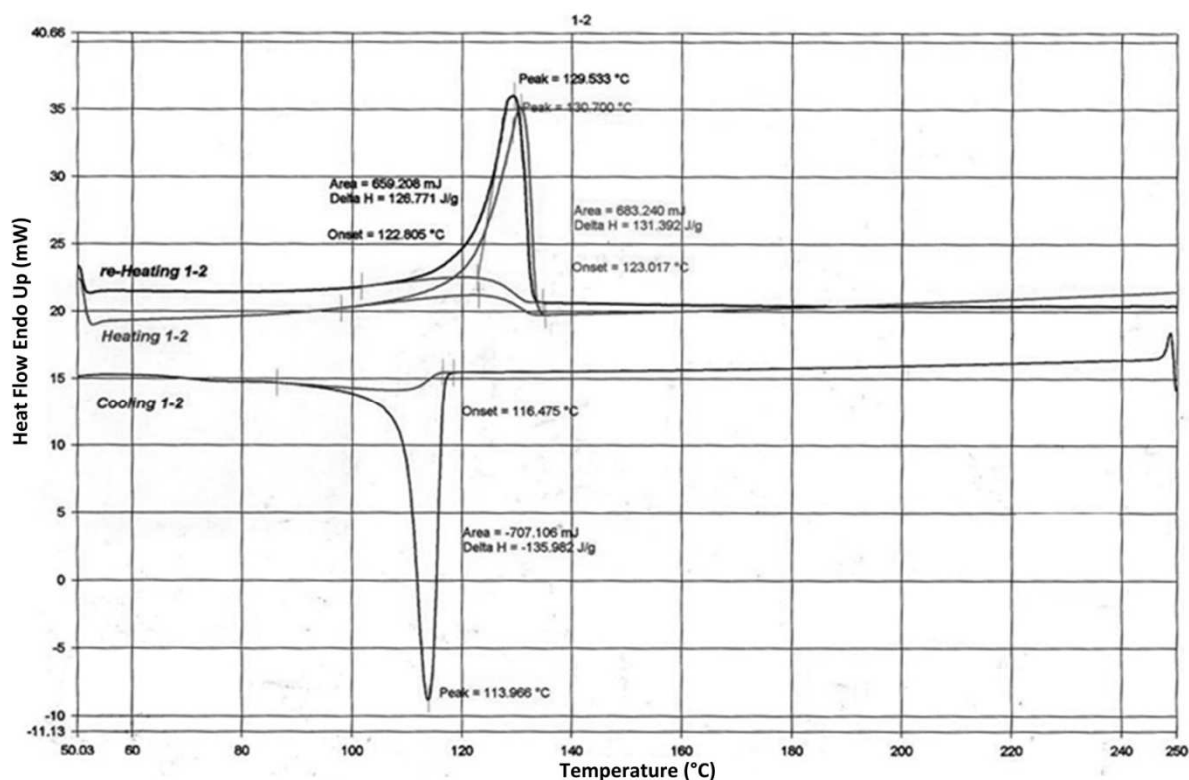


Figure 5: DSC thermogram of the PE100 compound containing the anti-rodent of Alchemix.

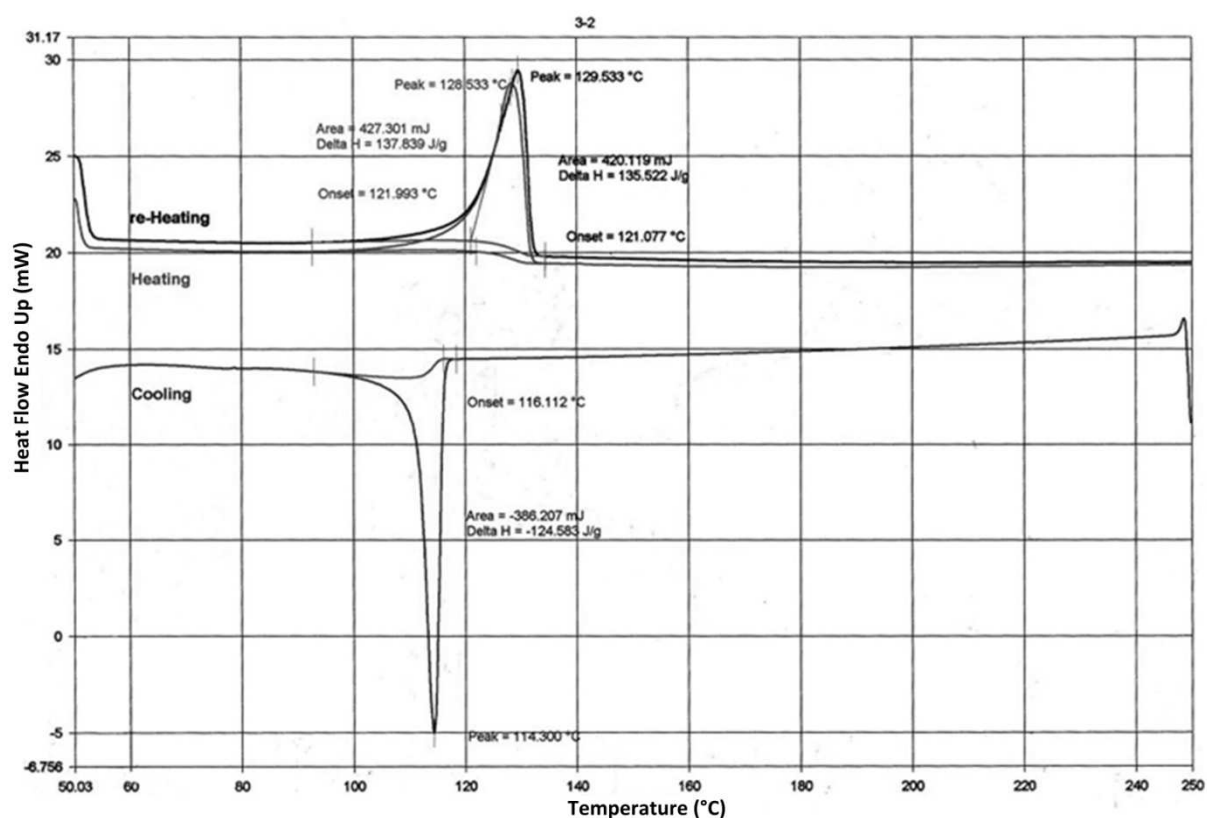


Figure 6: DSC thermogram of the PE100 compound containing the anti-rodent of Clariant.

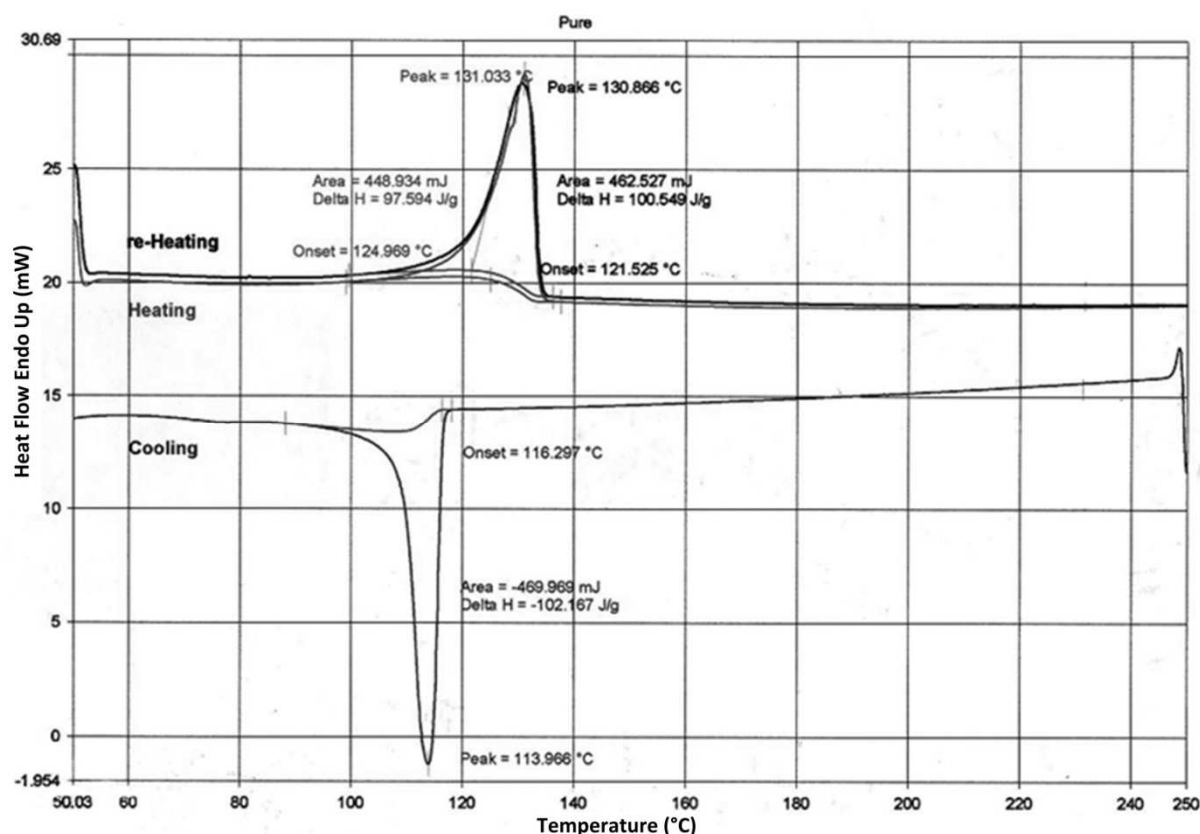


Figure 7: DSC thermogram of pure PE100 material.

Figure 8 shows a typical pattern of ductile failure (known as “parrot’s peak” or “fish mouth”) for high-quality polyethylene pipes in burst test. As shown in Figure 9, the burst test results of all the samples of anti-rodent added pipes show a ductile failure pattern, which confirms that the additive has no adverse effect on the pipes.



Figure 8: The pattern of ductile failure in polyethylene pipes

Hydrostatic tests of 100, 165 and 1000 hours were run on all anti-rodent-added and control PE 100 compounds. None of the samples failed

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within the specified test periods, showing the anti-rodent additives did not lessen the strength of PE pipes.

In order to find out the effect of the anti-rodent masterbatches on the oxygen induced thermal degradation behavior (OIT's) of the PE compounds in the melt phase, a series of OIT test was carried out on small pieces cut from the anti-rodent added and control pipes.



Figure 9: The pattern of pipe failures observed in burst tests for anti-rodent-added PE compounds

<http://jpst.ripi.ir>

Figure 10 shows the OIT results of anti-rodent-added pipe with sample code 4. As can be seen in Figure 10, after the appearance of the melting peak of the sample and concurrently switching the purge gas to oxygen nearly 70 minutes elapsed before second peak related to its degradation isotherm appears. This time period was measured 69.07 minutes for the control pipes (Figure 11). Thus, the OIT tests revealed

that the presence of the anti-rodent masterbatches in the pipes reduced their OIT's by around 20 minutes in comparison with the control pipe. According to the national standard for PE pipes, the OIT of the pipes is needed to be more than 25 minutes. However, the OIT's of the anti-rodent-added pipes are still twice as high as the standard limit, albeit decreased by adding the anti-rodent masterbatches.

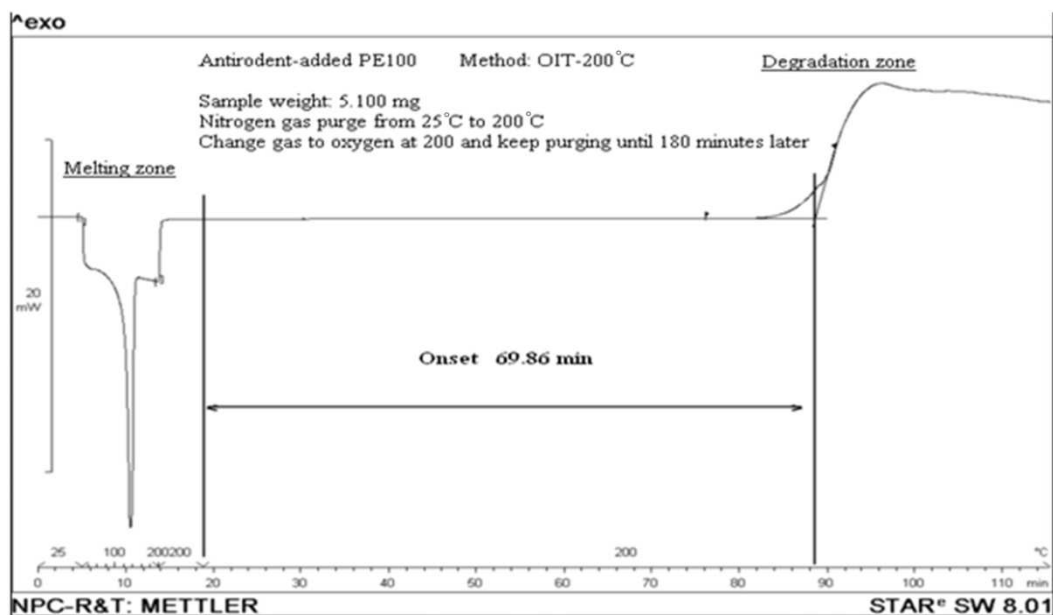


Figure 10: OIT result for the anti-rodent-added PE100 with sample code 4

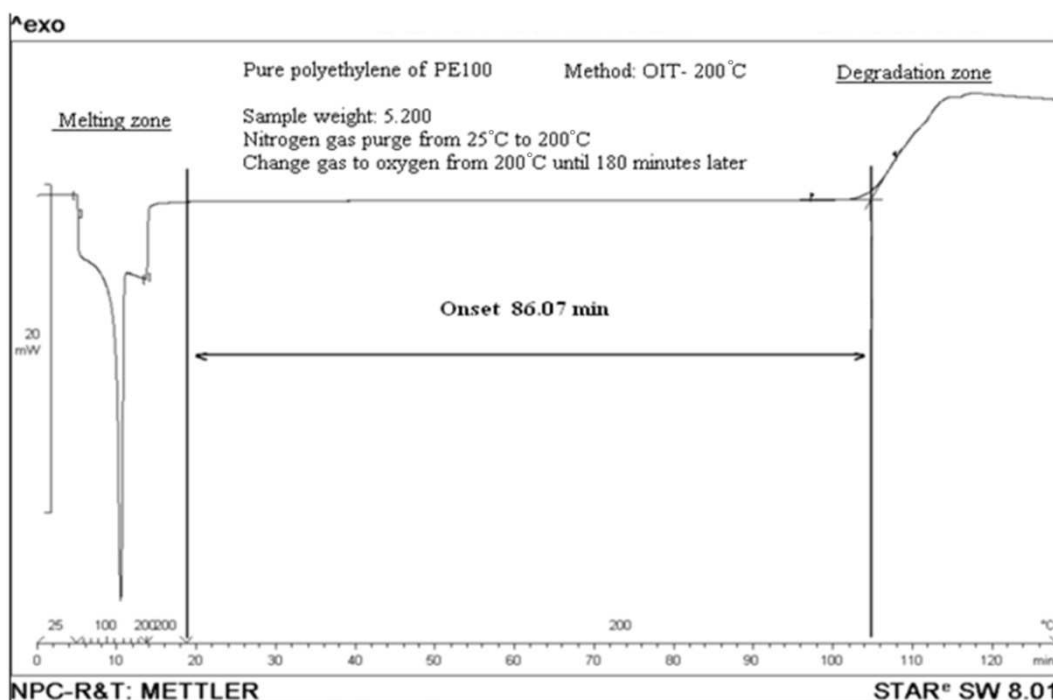


Figure 11: OIT result for pure PE100

## CONCLUSIONS

It was found out that the polyethylene pipes containing the anti-rodent additive successfully passed all the standard tests required for PE gas pipes. Although some small differences may exist between the test result figures of the anti-rodent added and anti-rodent free pipe samples, they are marginal and do not endanger the safety of the pipes.

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## REFERENCES

- [1] C-Tech.Corporation:  
website:www.ctechcorporation.com/pdf/Press\_Release.
- [2] C-TechCorporationnews.  
website:www.ctechcorporation.com/latest\_news.php
- [3] "A powerful aversive rodent repellent of Repela", Aversion Technologies website: [www.aversiontech.com/index.html](http://www.aversiontech.com/index.html).
- [4] ASTM D2513, "Standard Specification for Thermoplastic Gas Pressure Pipe, Tubing and Fittings", *American Society for Testing and Materials, West Conshohocken, PA*.
- [5] ASTM D638, "Standard Test Method for Tensile Properties of Plastics", *American Society for testing and Materials, West Conshohocken, PA*.
- [6] ASTM D1599, "Standard Test Method for Short-Term Hydraulic Failure Pressure of Plastic Pipe, Tubing and Fittings", *American Society for Testing and Materials, West Conshohocken, PA*.
- [7] ASTM F1473, "Standard Test Method for Notch Tensile Test to Measure the Resistance to Slow Crack Growth of Polyethylene Pipes and Resins", *American Society for Testing and Materials, West Conshohocken, PA*.
- [8] U. Anderson, "Which Factors Control the Lifetime of Plastic Pipes and How the Lifetime can be predicted". **2001**, *Proceedings of Plastics Pipes XI, IOM Communications*, London, 3-6 September.