Abstract for the 20th Plastic Pipes Conference
PPXX
September 21-23, 2020, Amsterdam, Netherlands

FINDING THE RELATIONSHIP BETWEEN NON DESTRUCTIVE TEST METHODS AND THE TENSILE IMPACT TEST ON PVC PIPES

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SHORT SUMMARY
In this paper the developments regarding two non-destructive tests for PVC pipes are given. The first is the Leeb hardness test , the second is the photo spectroscopic analysis of both the inside and outside of the pipe. Both tests are compared to the piping materials ductility determined with the tensile impact test, currently used to determine the quality of the PVC piping materials.

KEYWORDS
Polyvinylchloride, non-destructive technique, UV-Vis, hardness, tensile impact test.

ABSTRACT
More than 20,000 km of rigid PVC (or PVC-U) pipes are currently in use for the distribution of natural gas in the Netherlands. In this decade the majority of these pipes will reach their initially specified lifespan of 50 years. In the light of a possible replacement surge it is increasingly important to identify the actual material quality of these pipes.

To gain insight in the remaining quality of the PVC-U pipes in the Dutch gas distribution net, a so-called Exit Assessment program was started in 2004. In this program the quality of existing PVC-U material is determined by measuring the brittle-to-ductile transition temperature of the excavated pipes with a tensile impact test. [1,2]. The ultimate goal for Dutch Distribution System Operators (DSOs) is to assess the remaining quality in the field without the need for removal of pipe segments by non-destructive techniques.

This paper gives insight in the correlation between photo spectroscopic measurements and hardness measurements on the ductility of PVC pipes, revealing insight in the usefulness of these non-destructive technique for PVC pipes.

INTRODUCTION
Almost 70.000 km of PVC (rigid PVC (PVC-U) and impact modified PVC (PVC-HI)) pipes are currently in use for the distribution of natural gas in the Netherlands, which accounts for 64% of the low pressure gas grid (up to 8 bar) [3] The Dutch Distribution System

Operators collectively evaluate the quality of their grid in a program known as the Exitbeoordeling. For this program representative pipe segments are randomly taken out of the natural gas grid all over the Netherlands. The pipe segments are collected and tested by Kiwa Technology to determine quality by means of ductility. This test involves the destructive testing of the pipe segments.

The aim of this research is to determine quality of PVC via an alternative non-destructive method. Non-destructive testing has considerable benefits:

- Distribution System Operators would no longer have to take out pipe segments from their grid to evaluate quality.
- No interruption of the gas flow is required for analyses.
- On all locations were the PVC network is exposed an analysis can be performed.
- In future, an inspection robot can evaluate the quality of the PVC from the inside of the grid.

Hardness and color are two material properties which can be determined using a non-destructive method. This paper describes the determination of the material properties for a number of PVC pipe segments and the comparison to the currently used destructive test-method, the tensile-impact test.

Failure modes

In order to assess the remaining quality of a product, it is important to look at its lifetime limiting failure mechanism. Failure data shows that spontaneous failure of the PVC pipes hardly occurs. The main cause of failure of PVC pipes is due to third party damage, followed by subsidence of the ground and inadequate installation [4] Failure caused by third party damage is often the result of excavation activities. During these activities the neighboring PVC pipe receives a lifetime-limiting impact followed by a gas leak. There are two different failure modes:

1) Brittle fracture;
2) Ductile fracture.

Although both failure modes are unwanted, it is important that the PVC pipes have the tendency to fail in a ductile way. A ductile fracture occurs at a higher energy compared to a brittle fracture and is therefore less likely to occur. Brittle fractures result in larger gas outflows. It is easier to stop and repair the gas flow from a ductile failed pipe [5] The quality of the PVC pipes is therefore assessed on the tendency of the material to fail in a ductile manner. The ductility of the material is determined with the tensile-impact test.

Tensile-impact test

During the tensile-impact test PVC specimens are broken using a high-speed tensile load (impact) introduced through a swinging pendulum. The resulting fracture mechanism is dependent on a variety of parameters of which the most important are:

- The impact; energy, direction and speed;
- The test specimen; material, shape, orientation;
- The environment; temperature.

The performed tensile-impact test is designed so that the impact can be considered constant. Both impact energy, direction and speed are constant. The test specimens shape is constant apart from the thickness, which is dependent on the wall thickness of the pipe. During the impact test, temperature is varied to alter the failure mechanism. At high temperatures the material is more likely to fail ductile. Opposite, at low temperatures the test specimen is more likely to fail brittle. With the tensile-impact test the temperature at which the failure mechanism shifts from brittle to ductile is determined, referred as the brittle-to-ductile transition temperature (Tbd). The lower the Tbd the better the ductility of the material and the better the material quality regarding impact resistance.

<u>Hardness</u>

The linear correlation between hardness and yield stress is described in [6] The yield stress in return is correlated to physical aging of PVC-U. This indicates the correlation between hardness and the aging of PVC materials. For the study described in [6], the hardness was measured using a Brinell hardness measurement. The Brinell hardness is acquired pressing a small sphere into the test specimen. This is a relatively slow type of hardness measurement. The current study intends to correlate the hardness with the materials resistance against an impact. During an impact a fast-mechanical load is applied onto the material, therefore also a fast hardness measurement is selected, the Leeb hardness. The Leeb hardness test uses a small sphere which travels towards the material at a high velocity and collides with the material. Due to the impact the sphere bounces from the material. The relative change in speed before and after impact is used to calculate the Leeb hardness. It is suspected that the fast impact hardness measurement is best to correlate the rapid cracking of material during impact.

<u>Color</u>

The color of a material is a property that is not directly linked to the mechanical properties of a product. Therefore, it is not obvious to determine the color of a PVC pipe to estimate its quality. There are however a variety of examples were a change in color indicates a change in quality. Two examples are given for illustrative purposes:

1. Like all polymeric materials, PVC has a limited resistance against weathering. It is therefore that the pipe producers apply a foil over the tube to protect the pipe between production and installation. Influence from unprotected exposure to direct sunlight has been studied and results in a discoloration of the material [5]. Indicating a correlation between unprotected weathering of the PVC pipe and a change in color.
2. Some polymeric materials whiten when the material is stressed by bending. When the load is continued, the product will fail at the white discoloration. The whiting of materials due to stress is known as ‘stress-whitening’. PVC is susceptible to stress-whitening. This also indicates a correlation between a change in color and a change in mechanical properties of the polymeric material.

Based on the two examples, a correlation between the color of the material and the materials ductility may be expected.

The color of a material is dependent on three parameters, namely:

A. The spectral characteristics of the light source;
B. The relative reflectance or transmission characteristics of the object;
C. The interpretation by the observer.

Parameters A and C are not dependent on the material itself. Therefore, only parameter B is of main focus. For completeness the intensity of the reflected radiation between 200 nm and 890 nm of the PVC pipes are determined by photo spectroscopy. Besides the visible light spectrum (between 380 nm and 740 nm) also a region of the ultraviolet and infrared light is measured.

EXPERIMENTAL

Tensile-impact test

The ductility of a PVC pipe is measured using the tensile-impact test. A tensile impact tester in accordance with ISO 13802 [7, 8] measures the amount of energy needed to fracture the PVC test specimen. For each excavated pipe a series of 30 test bars is machined distributed over the circumference of the pipe. During the test, climate is controlled using a temperature conditioner . Were each test specimen is struck by the pendulum at a different temperature, ranging from -25 °C to 47.5 °C. The test procedure is described in more detail in [9].

Leeb hardness

The Leeb hardness is determined just before the conditioned test bar is struck by the pendulum during the tensile-impact test. For the measurement a portable handheld hardness tester is used. The Leeb hardness is determined for all 30 test bars, thus also over the temperature range from -25 °C to 47.5 °C in [HL].

Photo spectroscopy

Before the tensile-impact test is performed, 10 out of 30 machined test bars are selected based on their orientation around the circumference of the pipe. The relative reflectance over wavelength is determined over a range between 200 nm up to 890 nm by using a spectrophotometer equipped with a 150 mm integrating sphere in accordance with ASTM E1331-15 [8]. The spectral reflectance at both the inside and outside of the test bars are measured.

The spectra are converted to the CIE-Lab color space assuming D65 illumination (daylight) and the CIE 1964 standard colorimetric observer (10°). This results in three coordinates; L* for the lightness from black (0) to white (100), a* for green (-) to red (+), and b* from blue (-) to yellow (+). Because the L*, a* and b* coordinates result in a three dimensional color space, it is difficult to indicate differences in color solely on these coordinates. Therefore a standard method is used, the CIEDE1976 , a mathematical method to determine the distance between two points (ΔE_{76}) in the color space by deriving the Euclidean distance. This method is used to determine color differences within the excavated pipe, f.i. the color difference between the outside and inside of the pipe.

RESULTS

Typical results from the Tensile-impact test

Figure 1 shows a typical result from the tensile-impact test of a PVC-U pipe were the temperature is set against the fracture energy. Each datapoint represents a broken test bar. From the tensile-impact test a variety of material properties are obtained, namely; the brittle-to-ductile transition temperature (Tbd), the mean energy of a brittle failure (Eb) and the mean energy of a ductile failure (Ed). The ductility of the piping material is based on the Tbd. The lower the Tbd the better the material quality regarding impact resistance. A low transition temperature represents a good PVC material and a high one a poor PVC material.

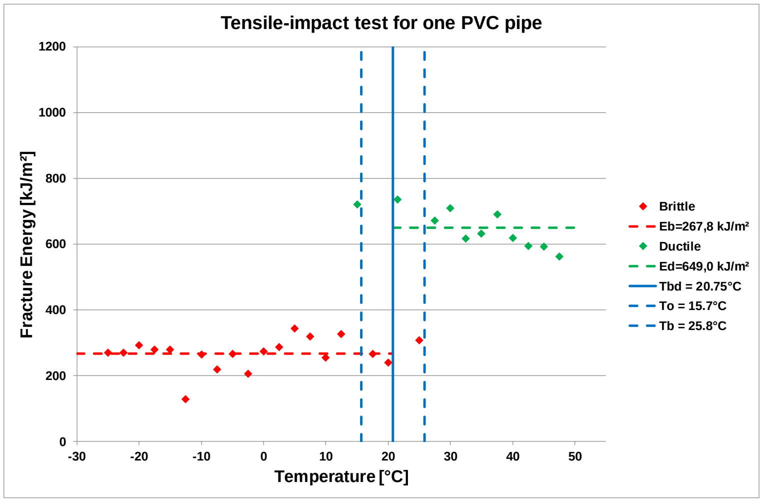

Figure 1. Brittle-to-ductile transition temperature for a typical single PVC pipe measurement.

In Figure 2 the histogram shows the ductility of all individual measurements of the tensile-impact test from 249 excavated pipes grouped for PVC-U pipes (red) and PVC-HI pipes (blue). Both the PVC-U pipes and PVC-HI pipes are normally distributed. Therefore, also the non-destructive test methods are separately evaluated for both types of PVC. The histogram shows the good PVC-U pipes having an equal ductility compared to the poor PVC-HI pipes. Which is very useful information concerning the replacement policy of old PVC-U pipes.

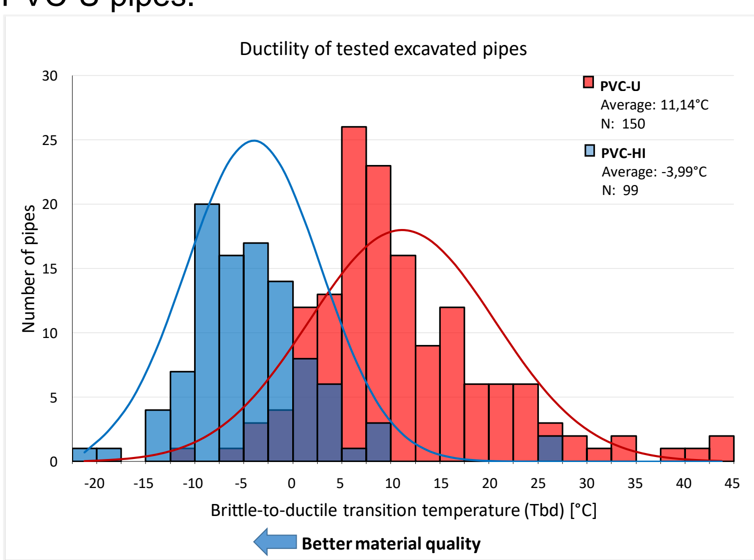

Figure 2. Histogram of the brittle-to-ductile transition temperature of excavated PVC pipes.

Correlating the Leeb Hardness

Figure 3 shows a typical result from the hardness test. Each datapoint is the hardness of a conditioned test bar just before it is struck by the pendulum of the tensile-impact test. Figure 3 shows the dependence of the Leeb hardness to temperature. Two parameters are derived from the measurement, namely; the slope of the linear regression curve and the height of the linear regression curve (chosen at 10 °C, average soil temperature in the Netherlands). Both parameters are used for comparison with the Tbd, see Figure 4.

The results of the Leeb hardness test compared to the ductility of the piping material is shown in Figure 4. There appears to be a cloud of points, no clear relation between the Leeb hardness measurements and the ductility of the pipes can be observed. This also applies when both the slope and height of the linear regression curve of the hardness measurements are compared to the mean energy of brittle failure and the mean energy of ductile failure, date not shown in this paper.

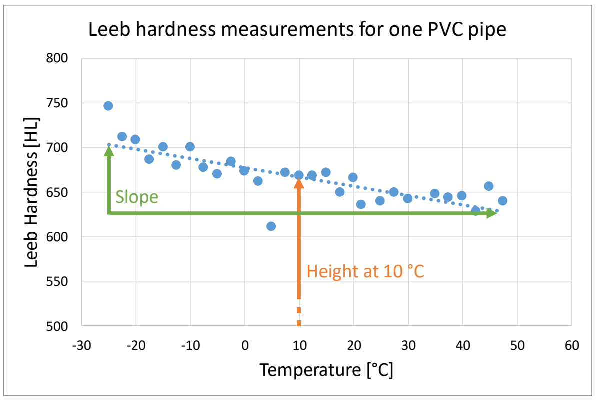

Figure 3. Leeb hardness of conditioned test bars over a temperature range for a typical single PVC pipe measurement.

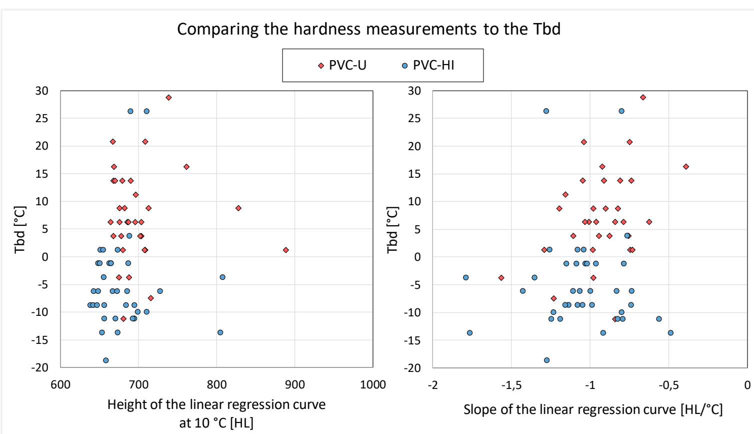

Figure 4. Results obtained from the Leeb hardness measurement compared to the brittle-to-ductile transition temperature of the respective PVC pipe.

Correlating photo spectroscopy

Figure 5 shows the spectra taken from the test bars of one PVC pipe. The data is used to determine the CIE-Lab color space coordinates (L*, a* and b*) and the distance between the average color space of the inside of the pipe and the outside of the pipe derived via CIEDE1976. The results of the spectra in Figure 5 are shown in Table 1. The spectra are also processed using principal component analyses by software program Unscrambler version 10.5, as a tool to obtain a numerical value for the differences between the spectra (the uncorrelated data). The principal components, the color space coordinates and the CIEDE1976 are used for comparison with the Tbd.

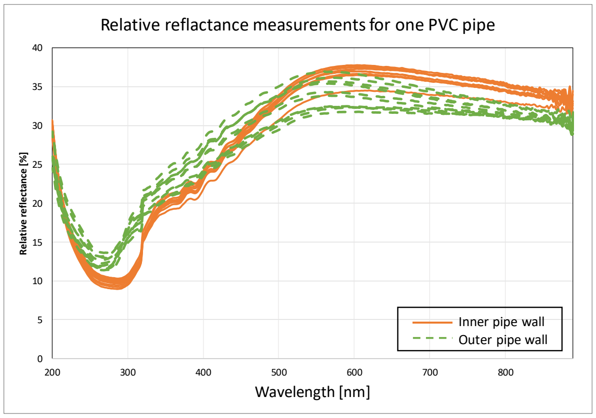

Figure 5. The spectra obtained for a typical single PVC pipe measurement.

Table 1. The processed spectra of Figure 5 result in the color space coordinates L, a* and b* by using the D65 illumination and the CIE1964 standard observer. The distance between the color space coordinates of the inside and outside of the pipe is determined*

	Average of 10 measurements L*	a*	b*	ΔE_{76}
Inner pipe wall	66.01	-1.20	9.64	4.35
Outer pipe wall	64.45	-1.31	5.59	

No clear correlation between the collar space coordinates (L*, a* and b*) and the ductility of the pipe can be observed. (Data of this correlation is not shown in this paper). One can question the use of the CIE-lab coordinates because they are the translation of the human interpretation of the spectrum. For instance; a clear difference between 200-350 nm (UV-light) cannot be observed in the CIE-lab coordinates. This is overcome by analyzing the principal components, which are based on the entire measured spectra.

In Figure 6 the principal component analysis of the inner pipe wall photo spectroscopic data is depicted. This includes the 10 measurements per excavated pipe. A clear distinction between PVC-U pipes and the PVC-HI pipes over principal component 1 can be observed. This is as expected because the difference between both pipes can clearly be observed by human eye, where PVC-U pipes are whiter compared to the more yellow PVC-HI pipes.
From the 10 photo spectroscopic measurements of the inner pipe wall per excavated pipe the average principal component 1 is derived. In Figure 7 the average principal component 1 is depicted against the ductility of the studied pipes. A clear correlation between the average principal component 1 and the ductility of all pipes can be observed, were the higher the principal component 1, the higher the Tbd, see black dotted line. However, the PVC-U pipes and PVC-HI pipes need to be evaluated separately since they form two

different populations in the Tbd results, see Figure 2. When considering just the PVC-U or PVC-HI pipes, no clear relation can be observed. It should be noted that the sample size is limiting. Also, the principal components of the outer pipe wall photo spectroscopic data are studied, which did not result in additional observations.

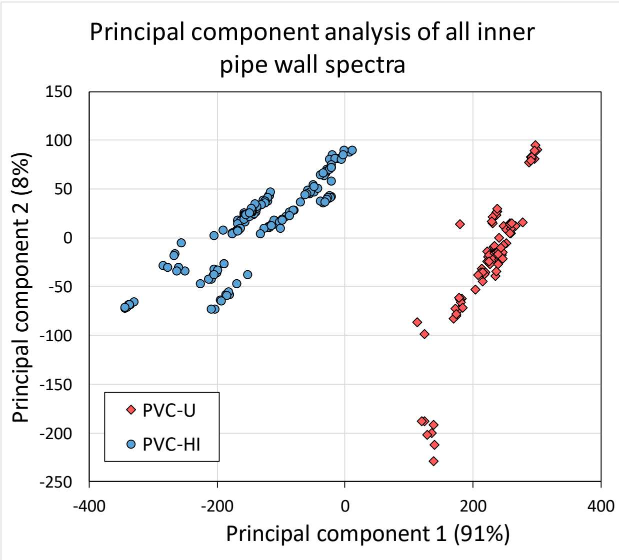

Figure 6. Principal component analysis off all spectra obtained from the inner pipe wall measurements. Per pipe, a total of 10 measurements is shown.

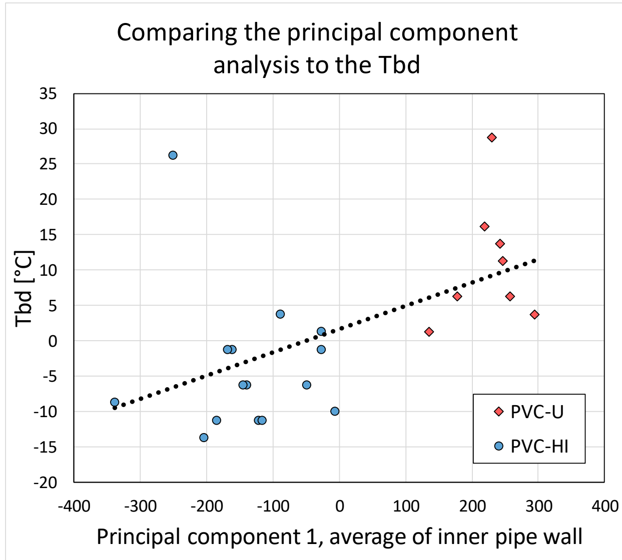

Figure 7. Per pipe, the average of principal component 1 compared to the brittle-to-ductile transition temperature. Each average is derived from the 10 respective measurements as shown in Figure 6.

Possibly not the color itself but a color difference over a pipe can indicate a change in material properties. For instance, a difference in color between the inside of the pipe and the outside of the pipe indicates influence of the environment on the pipe, either due to the transported medium or the conditions of the soil or the environment between production and installation, possibly weathering. The derived CIEDE76 values comparing the inside of the pipe to the outside of the pipe are compared to the ductility of the pipe in Figure 8. No clear relation can be observed. It can also be argued that not the color difference between the inside and outside of the pipe, but the color difference over the outside of the pipe or over the inside of the pipe should be considered. This has been done but did not result in a clear relation to the ductility of the pipe. (Data of this correlation is not shown in this paper).

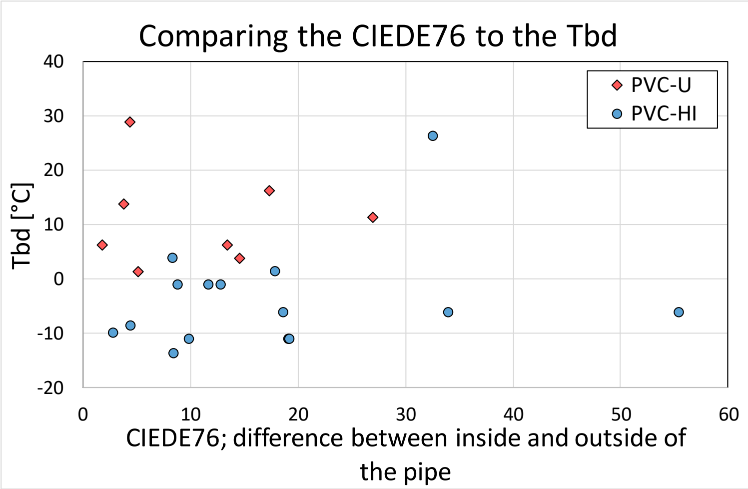

Figure 8. The ΔE76 obtained from spectra measurement compared to the brittle-to-ductile transition temperature of the respective PVC pipe.

Multivariant statistics

The population of pipes tested using the Leeb Hardness measurement is sufficient for Multivariant Statistics. Both parameters obtained from the hardness measurement (slope and height of the linear regression) and the sample thickness are used to build a model to predict the ductility of the material. This is studied by Multivariant Statistics using software program Unscrambler version 10.5 and SPSS version 26. Although the correlation of the model exceeds the correlation of the individual measurements to the materials ductility, with a root mean square of 0.26, the correlation is insufficient to adequately predict the materials ductility. The population size of pipes tested using photo spectroscopy is insufficient for effective Multivariant Statistics.

DISCUSSION

During this study, randomly selected pipes are excavated by the Dutch distribution operators and studied by Kiwa Technology. Therefore, large varieties within the population are present, for instance; duration of use, diameter of the pipe, pipe producer, soil type, ed. These varieties do not influence the materials ductility, since a well-defined normal distribution is obtained. However, the varieties can shade possible correlations between the ductility of the pipe and the non-destructive test methods. Although the varieties are known, the current population for the color measurements is thus small that no subdivision could effectively be studied.

This paper only elaborates on the correlation between a single non-destructive test method and the ductility of the pipe. Although no clear relation between a single non-destructive test method and the materials ductility could be observed, it is reasonable to assume that a combination of parameters can exhibit a relation. These parameters can be derived from multiple non-destructive testing techniques and data known by the distribution operators. The data can then be assessed using Multivariant Statistics to generate a ductility predicting model.

Because both increasing the subdivisions in the current population or increasing the amount of parameters requires an increased dataset, the current research is continued to expand the database.

CONCLUSION

The difference in material quality of excavated PVC pipes is studied by determining the materials ductility. It is shown that the good rigid pipes have an equal ductility compared to the poor impact-modified pipes.
The temperature dependence of the Leeb hardness measurements on the PVC specimens is shown. Despite the extended dataset, no relation with the materials ductility can be observed.
The photo spectroscopic measurements can clearly distinct rigid PVC pipes and impact-modified PVC pipes. Also, a relation between the principal component analysis of photo spectroscopic measurements and the materials ductility is shown. However, this relation is based on the entire dataset and does not distinct between the PVC type. Considering solely rigid PVC or impact-modified PVC, no clear relation can be observed.

The described study is ongoing, and this paper shows the intermediate results. Currently the dataset is expanded to also effectively analyze the photo spectroscopic dataset with multi variant statistics.

ACKNOWLEDGEMENT

The authors would like to thank Netbeheer Nederland and all cooperating Dutch Distribution System Operators in the Netherlands for their support in conducting the research on plastics pipe materials. Thanks also go to Jolanda Brugman and Paul Stens for carefully carrying out the many experiments.

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