Development of a "Nol Ring" test to study polyethylene pipe degradation and its implementation on field house connection pipes

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1 INTRODUCTION

The developpement of polyethylene production processes and the increase of industrial production capacities in the fifties led to generalize the use for distribution of gaseous fuel, drinking water and industrial applications.
The success of polyethylene for this uses is due to its advantages: low cost, versatility in setting up, weldability, flexibility, density, chemical resistance and toxicology safety.
In Europe, since 2003, premature brittle failures of polyethylene house connection pipes occurred on some water networks using chlorine dioxide as disinfectant. These failures are due to the appearance and developpement of longitudinal cracks in the internal pipe wall after a very short time of use regarding to the life time expectancy of polyethylene pipe which has to be over 50 years for conveying water at 20°C.
Thus, the durability of these networks using disinfectants appears to be an economic and strategic parameter for the water distribution companies. The use of polyethylene pipes could become questionable for the networks, in specific operating conditions where premature failures have already happened.
Some recent studies [1] [2] and [3] have shown that the most commonly used disinfectants Chlorine, Chloramines, sodium hypochlorite and Chlorine dioxide particularly oxidise the inner surface of polyethylene pipes with a significant depletion of antioxidants as result PE is embrittled. Micro-cracks appear and coalesce into large cracks, which can propagate through the pipe wall and induce brittle failure.
This effect, recently revealed, has to be evaluated and taken into account in order to estimate the durability of the polyethylene water networks. In that way, the Laboratoire National de Métrologie et d'Essais (LNE) has developed a new test called "Nol Ring" that has been implemented on 115 samples, picked up within several Suez-Environment's drinking water networks over 7 different countries, representing different PE grade, nominal pressure, water quality and operating conditions.

2 DEVELOPPEMENT OF THE NOL RING TEST

To assess the effect of disinfectants on polyethylene pipes, several data are required like for instance a precise pipe identification (classification, raw material, extrusion, date of production), its operating conditions (disinfectant, operational duration, history) and the characterization of its potential damages (internal or external).
Usually, the shortest test commonly used to identify the mechanical properties is the tensile test according to the ISO 6259-1/3 standards [4,5]. In that case, dog bones specimens are cut from the pipe wall parallel to the pipe axis, that is not representative of the stress encountered in operating conditions. Consequently, this technique does not appear well adapted to reveal

damages due to cracks, parallel to the pipe axis as observed for brittle failures. Other test could be conducted like the hydrostatic pressure test that reproduces the circonferial stress on the internal pipe wall but the analytical time is too long and expensive. Thus, the development of the test method called “Nol Ring” has been oriented towards a more realistic test than tensile test but easier to carry out and cheaper than common pressure tests. It has been inspired from the ASTM standard D 2290-04 [6] adapted for characterizing the degradation evolution of polyethylene pipes.
Ring specimens cut from the pipes are subjected to hoop (circumferential) stress similar to the one induced by pressure on the internal pipe wall in operating conditions. In order to compare the two ways of loading pipe walls, a correlation between Nol ring test results and Quick burst test results according to ASTM D 1559-2005 [7] has been searched.

2.1 DESCRIPTION OF THE TEST METHOD

The ASTM D 2290-04 describes a specific tensile test for determining the apparent hoop tensile strength applicable to thermosetting resin pipes and extruded pipes. It is indicated (Note 1, Procedure C) that similar results have been obversed in comparison with Quick Burst Test (ASTM D 1599) for 4 to 8 inch pipes of polyethylene and polybutylene.After enquiry, it has been impossible to find these tests results or works on this topic.
From the standard description, the developed Nol Ring test has been adapted to small diameter pipes (from 25 to 63 mm) as it is commonly used for house connection networks where brittle failures occur.

Procedure

Rings of 20-mm (See figure.1) are cut normal to the pipe axis with parallel sides. Two reduced areas are obtained by drilling two holes of 6.5 mm diameter, located firstly where the minimum wall thickness is measured and secondly at its 180° opposite side. The specimen is mounted in a split cylinder where the reduced sections are located in front of the split. The diameter of the cylinder is slightly lower than the inside diameter of the specimen. Then, the test specimen is loaded through a test fixture as shown in figure 2. The supports for holding the tension test fixture are self-aligning and attached to the fixed and movable member of one universal testing machine.
The drive mechanism of this device imparts a uniform and controlled velocity and then applies a tensile strength on the half cylinders perpendicular to the pipe axis. The test specimen is subjected to a (hoop) circumferential stress especially in the clearance between half cylinders. The test is carried out until failure. The stress at break is similar to the circumferential pressure at failure for pipes subjected to pressure test. The curve “load versus displacement of the crosshead” is recorded.

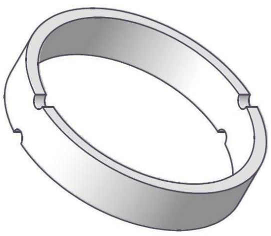


Figure 1: test spécimen

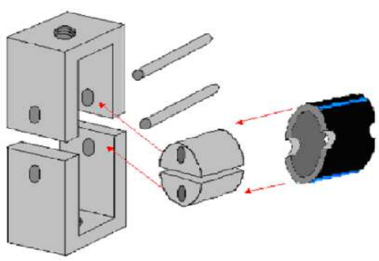


Figure 2: Nol ring test fixtures

2.2 PRELIMINARY TESTS

In order to implement this new test and fit relevant parameters, some measurements have been performed on specimens cut from new potable water pipes, as detailed below:

- PE 80 Nominal outside diameter: 40 mm, nominal wall thickness: 3.7.
- PE 80 Nominal outside diameter: 32 mm, nominal wall thickness: 3.0

The test speed is 12,7 mm/min. Five tests specimens are usually taken per pipe. The chart "stress versus elongation rate" is presented figure 3.

The sample looks like a wide dog bone specimen of 3 mm length in any case. This value has been chosen as the initial length. The elongation rate is taken here as the ratio of this length (3 mm) to the crosshead displacement. Firstly, a necking occurs at the minimum thickness restricted area until reaching the yield point for this area. As the test run, the yield point for the thickest restricted area appears. Then, the plastic deformation of the two parts of the ring between the half cylinders is observed, followed by a raise of the stress and at the end the break of the specimen.
This one is usually due to a crack that starts from the inner side of the thinnest ring part and propagates until failure. In figure 4, specimens of new pipes after test are presented.

Some other tests on field pipes coming from damaged networks or pipes with failures have also been performed [8].

All these preliminary tests have shown that the specimen achievement is controlled and the test repeatability, evaluated from the aspect of tensile curves and standard deviation of the main characteristics, ensured.

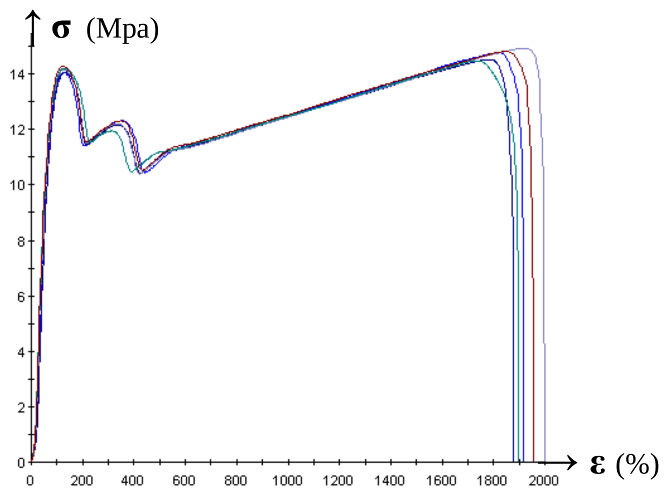


Figure 3 . Nol Ring test : chart stress versus elongation rate for five specimens cut from a new pipe new pipe

Figure 4 .View of two specimens cut from a new pipe after Nol ring test

2.3 Interpretation of Nol ring tests results and comparison with tensile test results on specimen cut along pipe axis

In addition to the preliminary Nol Ring tests mentioned above, some complementary experiments have been performed on pipes at different levels of degradation: from new pipes to pipe coming from potable water distribution networks where multiple brittle failures have already occurred.

The tests results have shown that two characteristics are relevant for the interpretation of the pipe degradation:

- the surface aspect of the specimen inner wall after the test (viewed without magnification)
- the elongation at break

Before the Nol Ring test, the specimen inner surface is black, smooth and exempt of scoring or cracks.

The pipe state can be classified according to five different degradation levels as described below,

- [0] - No modification of the surface aspect. The break starts from the inner side of one restricted area. The elongation at break is high and always ≥ 1500%. The stress at break is always higher than the stress at the second yield point.

- [1] - A light bleaching appears and sometimes a weak corrugation can be observed. The other characteristics are same as [0].

- [2] - Bleaching, scores and corrugation of the surface are observed. The break starts from the inner wall of the pipe, on one aged restricted area. The elongation at break is lower than previous ones and generally between 1100% and 1500%. The stress at break is often higher than the stress at the second yield point.

- [3] - Bleaching, scores and corrugation of the surface are more visible. The break starts from the inner wall of the pipe and can be located between the two edges of the restricted areas. The elongation at break is low: between 150% and 1100%. The stress at break is often lower than the stress at the second yield point.

- [4] - a white line appears between the two restricted areas on the inner wall of the pipe. The failure is initiated from this line and the elongation at break is low. The second yield point is not observed.

2.3.1 Testing results

Tests according to the ISO 6259-1/3 standards (dog bone specimen cut from the pipe wall, in parallel to the pipe axis) have also been carried out on the same samples.

Both results (Tensile and Nol ring) are presented in Table 1 with associated Nol Ring classification. Each result corresponds to the mean of 3 individual values.

The comparison between tensile curves (Nol Ring and ISO 6259-1/3 standard) is detailed in table 2 with the Nol ring classification and the surface aspect observed on the test specimen.

Table 1 highlights that all the mechanical characteristics determined according to ISO 6259-1/3 standards complies with the general requirements for new pipes (15 Mpa for the stress at the yield point and 500% for the elongation et break).

Table 1 : Comparison of Nol ring test and tensile test on specimen parallel to the pipe axis

Pipe reference Nominal dimentions (diameter x thickness) (Field samples)	**Results of Tensile test according to ISO 6259-1/3 standards**		**Results of the Nol Ring test**		
	σ Stress at the yield point MPa (standard deviation)	**ε elongation at break %** (standard deviation)	**σ Stress at the first yield point MPa** (standard deviation)	**ε elongation at break %** (standard deviation)	**Nol ring Classification**
Sample A (25 x 3)	**18.2** (0.4)	**718** (39)	**20.2** (0.5)	**1950** (10)	**[0]**
Sample B (25 x 3)	**21.8** (0.1)	**594** (61)	**22.6** (0.3)	**1680** (60)	**[1]**
Sample C (25 x 3)	**19.4** (0.2)	**635** (12)	**20.7** (0.2)	**1380** (40)	**[2]**
Sample D (25 x 3)	**21.6** (0.6)	**522** (127)	**21.4** (0.4)	**355** (206)	**[3]**
Sample E (25 x 3)	**21.9** (0.5)	**608** (93)	**22.0** (0.3)	**106** (2)	**[4]**

The "stress vs strain" curves obtained by the ISO 6259-1/3 method are not suitable to characterize polyethylene pipe degradation. Indeed, all the tested pipe specimens comply with the standard requirements whereas no clear differences have been noticed between a non degraded pipe (Sample A, level [0]) and a pronounced (Sample C, level [2]) or severe degradation (Sample E, level [4]) of the pipe specimen.

On the contrary, the Nol Ring results show clearly a significative decrease of the elongation at break according to the degradation level of the tested pipe.

The Nol Ring classification based firstly on the surface aspect is fully in accordance with the elongation at break : the brittleness revealed by a low value of the elongation at break corresponds to scoring and corrugation of the inner pipe wall between the restricted areas after testing.

So, with the developed Nol Ring test it is possible to establish a continuous range of degradation levels according to the specimen aspect after testing and the obtained mechanical resistance values (stress and strain) from the Nol Ring tensile curves.

These results show that the Nol ring test is more relevant to caracterize pipe damages than the standard tensile test according to ISO 6259-1/3.

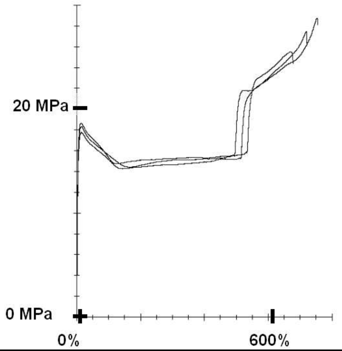
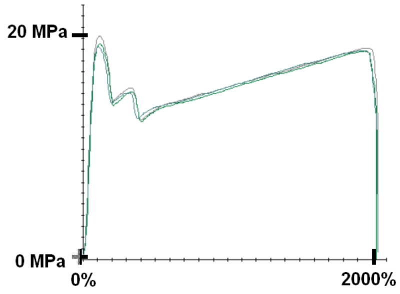
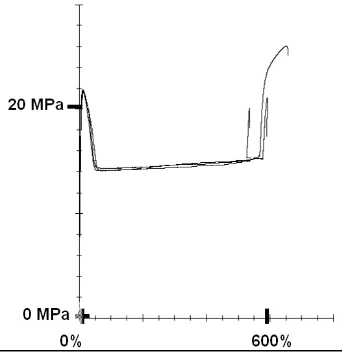
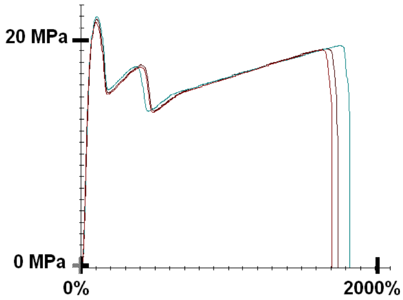
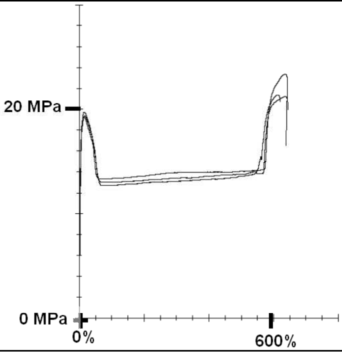
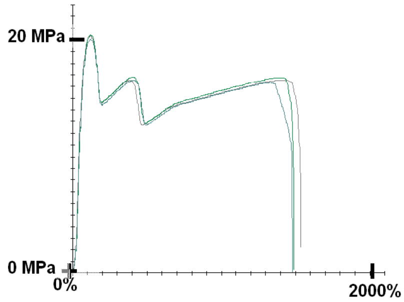
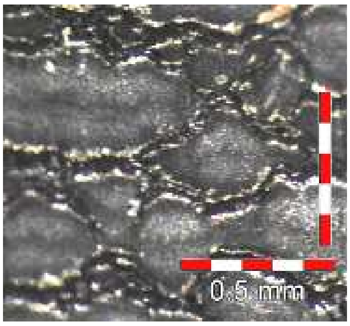
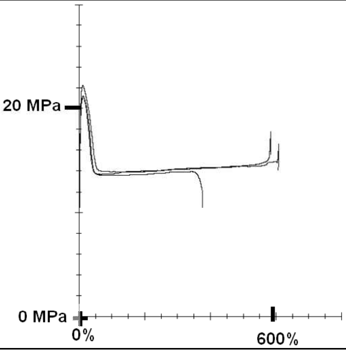
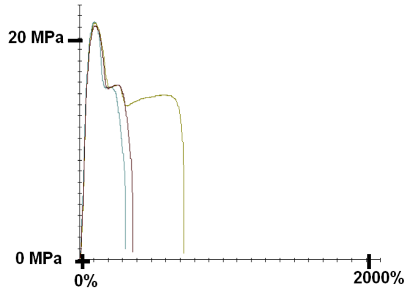
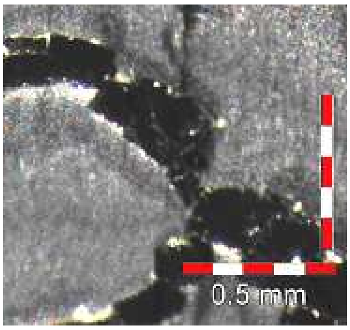
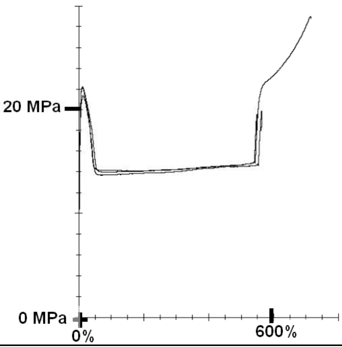
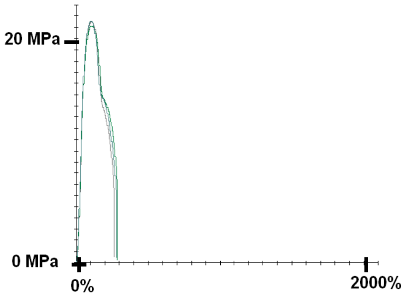
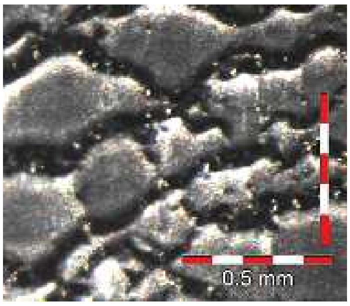
Nol ring Category (Nominal dimensions)	**Tensile test according to ISO 6259-1/3 Stress/strain curves**	**Nol ring test Stress versus elongation rate**	**View of the inner wall of the specimen afterNol ring test** ↕ direction of the tensile strength
[0] Sample A (25 x 3)			
[1] Sample B (25 x 3)			
[2] Sample C (25 x 3)			
[3] Sample D (25 x 3)			
[4] Sample E (25 x 3)			

Table 2 : Comparison between tensile curves (Nol Ring and ISO), Nol Ring classification and surface aspect of the specimen after Nol Ring test

2.4 VALIDATION OF THE PRINCIPLE OF THE NOL RING TEST: COMPARISON BETWEEN NOL RING TESTS RESULTS AND PRESSURE TESTS RESULTS.

2.4.1 Principle of the validation

Nol Ring test results have been compared to Quick burst test to verify if the hoop stress generated by the Nol ring is similar to the internal hydrostatic pressure

The Quick burst test method, defined according to ASTM D 1559 – 05 procedure A, establishes the short-time hydraulic failure pressure of thermoplastic pipes by increasing the pressure uniformly and continuously until the specimen fails. The time to failure shall be between 60 and 70 s.
The Nol Ring test subjects the test specimen to a hoop (circumferential stress), which, between the restricted areas, is quite similar to the stress induced by internal pressure.
From the Nol Ring results, the corresponding hydraulic pressure has been calculated according to the following equation (1)]

$$P = \frac{20 \times e_j \times \sigma}{D - e_j} \quad (1)$$

When:

P = the internal pressure (bar);
e_j = the thickness of the pipe wall at the restricted area (at break or at yield point) mm;
D = the outside diameter of the pipe (mm);
σ = the hoop stress (MPa).

2.4.2 Results

The test has been carried out on new PE 80 drinkable water pipes with the following nominal dimensions (nominal outside diameter x nominal wall thickness):

25 x 3.0 – 32 x 3.0 – 40 x 3.7 – 50 x 4.6 – 63 x 5.8 (mm).

The results are presented in table 3.

Table 3 : Comparison between pressures calculated from tensile hoop stresses according to Nol ring test and failure pressures determined from the ASTM D 1559-05 standard

Pipe Dimensions (mm) (Nominal outside diameter x nominal wall thickness)	**Calculated pressure** (bar) **according to equation (1) from the results of Nol Ring test** (standard deviation)		**Failure pressure (bar) according to ASTM D 1599-05** (standard deviation)
	At the first yield point	**At break**	
Sample F **25 x 3,0**	**54,9** (0,9)	**51,1** (2,3)	**51,7** (0,2)
Sample G **32 x 3,0**	**40,0** (0,2)	**40,1** (0,6)	**41,5** (0,1)
Sample H **40 x 3,7**	**46,3** (0,3)	**40,1** (0,9)	**47,1** (0,1)
Sample I **50 x 4,6**	**51,4** (0,8)	**38,0 *** (3,4)	**49,6** (0,5)
Sample J **63 x 5,8**	**51,9** (0,6)	**37,8 *** (5,1)	**50,4** (0,2)

For samples I and J, the break occurs before the second yield point. The elongation at break is weak.

Table 3 shows that calculated pressures at the first yield point or at break from Nol Ring test are quite similar to failure pressures determined according to ASTM D 1559-05 standard. This indicates clearly that the pipe specimen during the Nol ring test is submitted to a hoop stress representative to the pressure effect in operating conditions.

2.5 IMPLEMENTATION OF NOL RING TEST ON FIELD: CHARACTERIZATION OF THE DIFFERENT LEVELS OF DEGRADATION FOR POLYETHYLENE HOUSE CONNECTION PIPES

A large sampling campaign has been launched in order to collect different samples with diversified characteristics such as different age, disinfection process, material origin, operating conditions,… Among these ones, 115 samples were analysed by the Nol ring test. Figure 5 illustrates the number of samples collected according to the country.

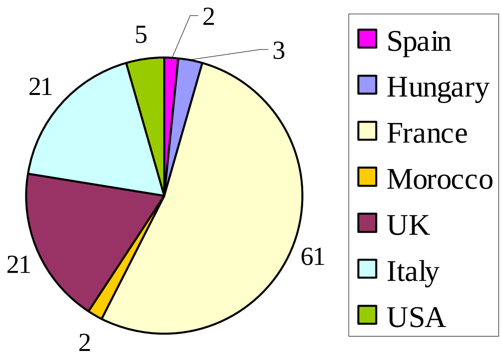


Figure 5. Sampling campaign for Nol Ring analyses according to the country

In order to understand the results, the samples were classified according to the previously defined Nol Ring classification:

- the [aspect] of the specimen after Nol Ring
- the elongation at break value, A%.

Figures 6 and 7 represent these rankings.

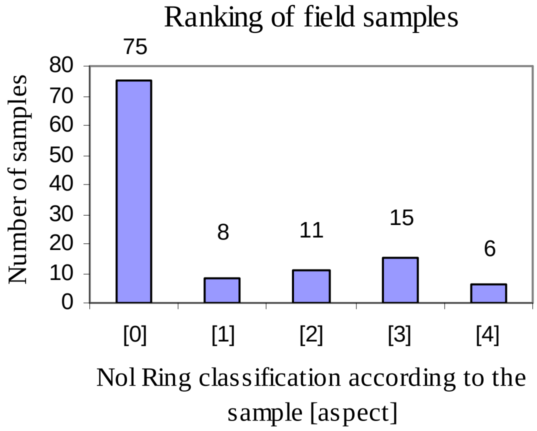


Figure 6. Screening according to the sample [aspect] after Nol Ring test

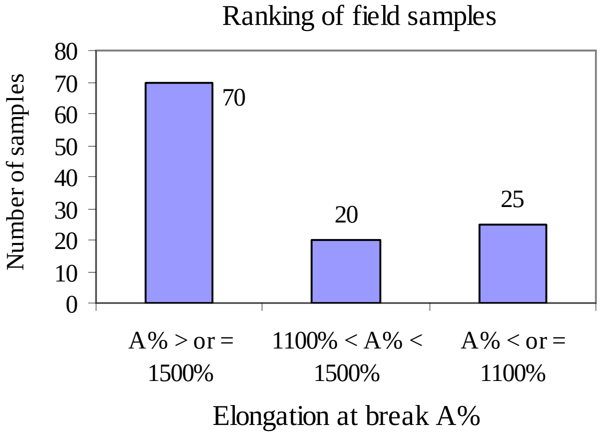


Figure 7. Screening according to the Elongation at break value obtained by the Nol Ring test

The classification with the elongation at break value highlights the mechanical resistance of the tested sample whereas the surface [aspect] remains subjective due to visual observations. However, as the parameter weight is unknown and very difficult to estimate, both criteria have to be taken into account for the degradation assessment. Consequently, a derivative index, called NRR ("Nol Ring Result"), was defined for field samples, as presented in Table 4.

Table 4 : NRR Index for the degradation assessment on field samples

Degradation Index	Green Level	Yellow Level	Red Level
NRR Index	[Aspect] < 2 AND A% > 1500%	[Aspect] = 2 OR 1100%<A% ≤ 1500%	[Aspect] > 2 OR A% ≤ 1100%

The three level classification, "Green, Yellow, Red", was developed for all degradation parameters identified as having an impact on the degradation process such as OIT and Carbonyl Index for the chemical oxidation processes, ODL (Overall Degradation Layer) for overall state, Hydrostatic Index for mechanical performance after hydrostatic pressure test and finally NRR, for the Nol Ring Test.
For this one, the "Green" level corresponds to none degradation for the sample [aspect] and the elongation at break value. The "Yellow level" and the "Red level" reveal respectively a moderate degradation and a high deterioration of the sample. One parameter, either the sample [aspect] or the elongation at break, is sufficient to classify the NRR Index in those categories.

With this new definition, Figure 8 illustrates the obtained results for the analysed samples.

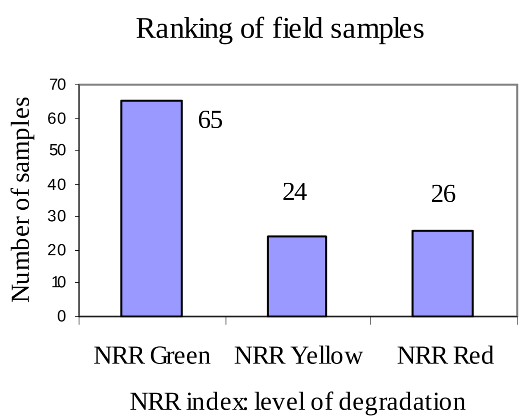


Figure 7. Screening according to the NRR index obtained by the Nol Ring test

It appears that this classification is more severe than the previous one. Indeed, it allows to see an intermediate degradation category taking into account both degradation criteria: the overall aspect and the mechanical performance with the elongation at break value. So, 18 samples were redefined in a degradation category, more severe than expected: 13 samples moved from the previous classification to the Yellow degradation level and 5 to the Red level.

Based on this classification, figures 8 to 10 show the repartition of the samples according to the country. It appears that the UK and Hungarian samples are mainly focused in the Green zone whereas the Spanish and Moroccan ones are in the Red Class. The major part of French samples are located also in the "no degradation" category althougth 1/3 of them presents moderate or high deterioration level. Italian samples can be seen in each category even if the main part is still in the higher degradation category.

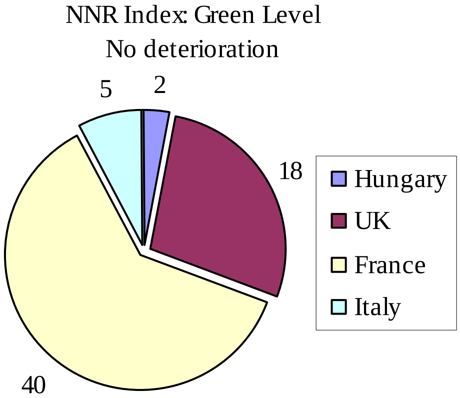


Figure 8. NRR results and repartition according to the country for not deteriorated samples

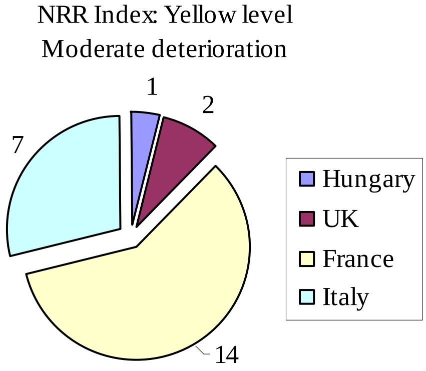


Figure 9. NRR results and repartition according to the country for moderately deteriorated samples

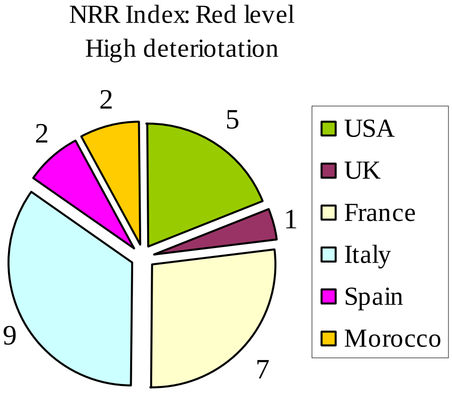


Figure 10. NRR results and repartition according to the country for high deteriorated samples

However, it has been noticed that the classification of the [aspect] criteria and the elongation at break parameter can differ according to the sample. Figures 11 to 13 present, by deterioration level the final ranking taking into account the influence of the disinfectant type.

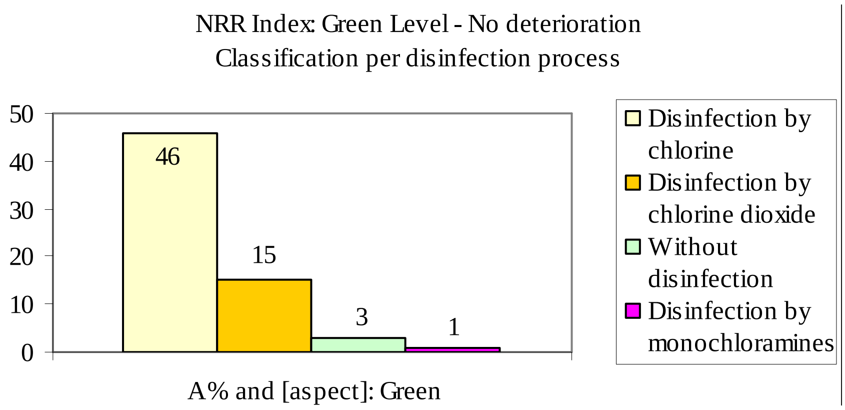


Figure 11. NRR results – Green Level / No degradation : repartition according to the disinfectant process

Among the 65 non degraded samples, the major part is concerned by samples disinfected by chlorine.

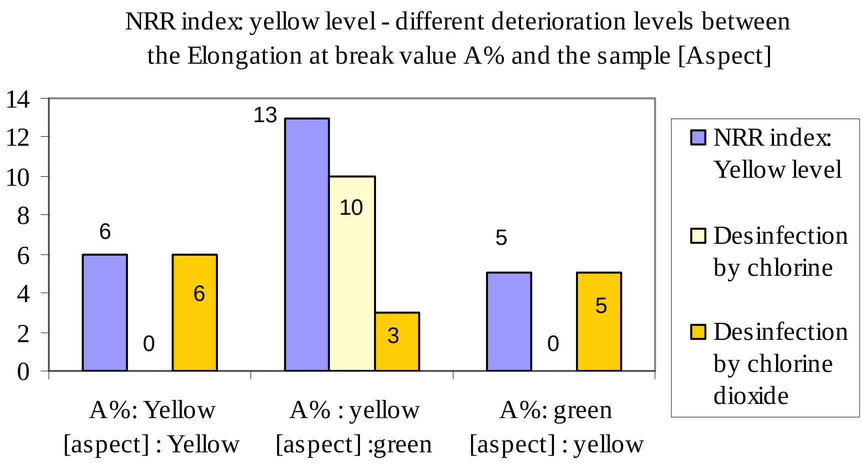


Figure 12. NRR results – Yellow Level / Moderate degradation : repartition according to the disinfectant process and distinction between [aspect] and A%

Contrary to that, with the Yellow and Red NRR categories, some differences are noticed. The most degraded samples, corresponding to the Yellow-Yellow and Red-Red classification, respectively [aspect] and A%, have been mainly disinfected by chlorine dioxide. The 4 samples with chlorine refer to the oldest specimens (30 years) in Spain and Morrocco, where raw water temperature is high.

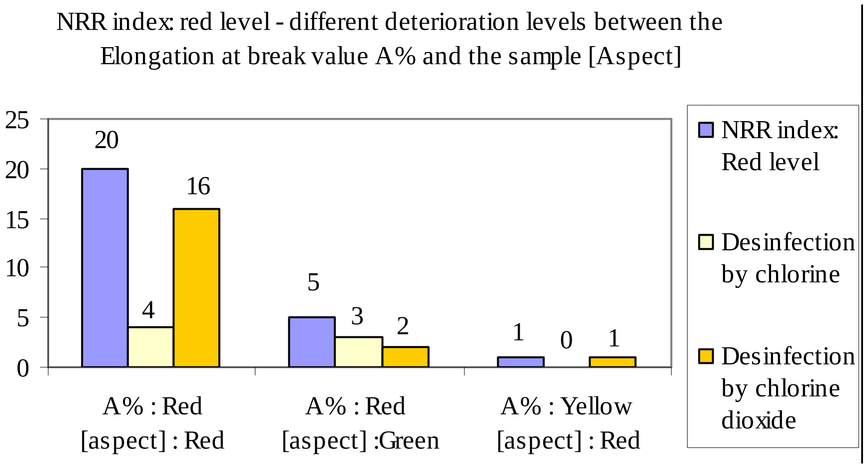


Figure 13. NRR results – Red Level / High degradation : repartition according to the disinfectant process and distinction between [aspect] and A%

The second category could join respectively the Yellow [aspect] / Red [aspect] to the Green A% / Yellow A% (6 samples) where the samples keep good or a moderate mechanical resistance with a green or yellow A % level. These ones could have a lower degradation state but always linked to the ageing by the oxidation process.

At the opposite, the last category with a "green" aspect value correlated to low or moderate elongation at break could refer to poor quality materials rather than oxidized samples by chemical ageing.

Consequently, the newly defined classification allows to identify the different levels of degradation and could highlight materials with poor mechanical performances.

3 CONCLUSION

A new characterization test, called “Nol Ring”, has been developed to assess the effect of disinfectants on polyethylene pipes. It has been designed to be simple to carry out and cheaper than classical hydrostatic pressure test, with a special care to the applied stress. This one has been validated by comparison to the Quick burst test method. It is well representative of a Hoop (circumferiential stress), which is quite similar to the stress induced by internal pressure found in water distribution networks. Then, a comparison with tensile test on specimens cut along the pipe axis have highlighted that only the Nol Ring test is able to identify damaged pipes by a significative decrease of elongation at break in accordance with damages noticed on the analysed sample. At the contrary, the usual tensile test did not significantly detect such degradation. Finally, the Nol ring test was implemented with the analyse of 115 field samples. A degradation index, called NRR, was defined to differentiate degraded samples according to the overall state of the sample and its mechanical performance. This newly classification allows to identify damaged samples by oxidation mechanism from those due to poor quality materials and constitutes one of the degradation indexes used for the diagnosis of the PE house connection network.

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