A study of the behavior of bi-oriented PVC exposed to ionizing radiation and its possible use in nuclear applications

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Received 19 October 2012
Accepted 22 February 2014
Available online 4 March 2014

1. Introduction

In the last years piping made with plastic materials have progressively replaced in many industrial applications metallic piping. Reasons for this replacement include lower costs in production, installation and maintenance and better resistance to chemical corrosion. In addition, underground piping networks made with plastic materials do not require cathodic protection or the use of other techniques to control the corrosion over the entire piping lifetime.

With respect to metallic materials, the major limitation for a wide use of plastic materials in industrial applications is related to low temperatures and pressure that these materials can withstand. However, typical applications of plastic piping extend to systems processing fluids at low temperature and pressure, and include firewater networks, sewer networks for collecting oily and contaminated waste water, process systems of corrosive fluids.

Among plastic materials, Polyvinyl Chloride (PVC) is by far the most widely used in industrial applications: in the last years the attention of PVC producers has focused on processes that, by the modification of the structure of polymers chains, aim at the enhancement of the mechanical properties thus allowing to broaden the field of possible applications. In the family of the

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modified PVC's, one of the most promising materials is represented by the so called bi-oriented PVC that, due to its peculiar orientation of the polymer chains in the bulk material, has a yield stress more than twice than unplasticized PVC, is less fragile and better withstands to shocks, thus overcoming the most common drawbacks that limit the use of PVC in many industrial applications.

It is worth questioning whether the enhanced mechanical properties of commercial bi-oriented PVC are affected when the material is exposed to ionizing radiation: this information is important to assess whether this material could be successfully employed as a possible replacement of metallic materials also in industrial applications where radioactive fluids are processed and ionizing radiation is present. In order to provide a first answer to this question, an experimental campaign was conducted with the aim of assessing the behavior of a commercial bi-oriented PVC when exposed to an intense field of β and γ ionizing radiation. In addition, preliminary contamination and decontamination tests of bi-oriented PVC in contact with a radioactive solution have been carried out. Our aims are to point out the possible applicability of PVC-BI in nuclear industry neglecting a deep insight into molecular alteration in order to focusing on the macroscopic behaviors.

1.1. The bi-oriented PVC

Polyvinyl Chloride (PVC) is a thermoplastic material that has been used since many decades for a wide numbers of applications. Approximately one half of the world's polyvinyl chloride resin manufactured annually is used for producing pipes and fittings for municipal and industrial applications (Rahman, 2007). Its light weight, low cost, good chemical resistance to corrosion and workability make it very attractive. With a chlorine content of 57%, PVC is much less dependent on the limited supply of gas and oil than other polymer products. Since the 70's the attention of PVC producers has focused on overcoming the major limits of the product, like fragility, low resistance to shocks and low crack propagation resistance. One interesting solution is the bimolecular orientation of PVC material. Bimolecular orientation is a process whereby, by applying mechanical deformation to a pipe previously extruded, a substantial modification of its mechanical properties is produced: mainly an increase of the allowable tensile strength, a better resistance to impacts, an improvement to creep, a better resistance to crack propagation, an increase of the Young module (Chauffoureaux, 1981). Orientation is achieved by drawing or stretching the PVC pipe previously extruded, under appropriate temperature and deformation speed conditions, so that a strain (deviation from originally formed dimensions) is induced in the bulk of the material to produce an alignment of the molecules in the direction of strain. After the orientation process, the pipe is cooled down quickly to ambient temperature. A number of patents have been development to produce Bi-Oriented PVC pipes. The pipes for the study were provided by GDS-SIRCI, Italian leader in the field of plastic materials pipes for building and industry applications. Bi-oriented pipes are produced with an innovative system that stretches and orients PVC pipe with air instead of water. This method was introduced in 2008 by Molecor, a Spanish technology development Company. The bi-orientation process is very sensitive to high temperature. If bi-oriented PVC is re-heated at a temperature greater than the glass transition temperature ($\approx 80\,^{\circ}C$), the bulk material completely looses the bi-orientation and returns to the original shape it had before the orientation process took place, i.e., its thickness approximately doubles, and its diameter halves. This phenomenon is known as 'reverse' and it is particularly critical since for a pipe it implies, along with unacceptable dimensional changes, the complete loss of enhanced mechanical properties. The bi-oriented PVC-U pipes for the experimental irradiation tests were produced in GDS-SIRCI Plant, Levate (BG) in Italy. Table 1 shows the pipe's formulation with concentration in phr (parts per hundred resin) and the trade name of each material.

Table 1
Bi-oriented PVC composition used for the tests.

Material	phr (parts per hundred resin)	Trade name
PVC	100	Ineos chlorvinyls – Norvinyl S6806
Organic stabilizer	3.2	Reagens-Reapak G-TU/1068
Calcium Carbonate	3	Nicem-Carb BCM 20
Pigment	0.8	Chimar-White CP r 31/08

The axial orientation coefficient λ_a, defined as the ratio of the lengths of PVC-Bi pipes and the pre-formed pipes,. is approximately 1.0 whereas the tangential orientation coefficient λ_t approaches 1.8 and is defined as:

$$\lambda_t = \frac{D1 - e1}{D2 - e2}$$

where $D1$ and $D2$ represent the mean external diameter of PVC-Bi pipes and pre-formed pipes respectively, $e1$ and $e2$ are the mean thickness of PVC-Bi and pre-form pipes. The preform pipes have a nominal diameter of 77.6 mm and nominal thickness of 7.6 mm, so the pipes after the stretch of 1.8 have a diameter of 140.1 mm and a nominal thickness of 3.9 mm, in accordance to ISO 16422 for Pressure Class 16. The preform pipes are extruded with an Argos 93 Cincinnati Battlefield extruder; the molecular orientation is achieved by applying Molecor Tecnologia patent's process conditions.

2. Experimental procedure

2.1. Numerical simulation

In view of the possible application of bi-oriented PVC in nuclear plants, a preliminary numerical simulation was carried out. The objective of the simulation was to assess the dose on the pipe wall due to the contact, over a specified time period, with a stream containing radioactive nuclides, with a specified concentration. In such a way it is possible to compare the effects that are expected in a possible industrial application, when the pipe, during its entire lifetime, conveys radioactive fluids, with those resulting from the irradiation tests that have been experimentally carried out.

The first assumption is that the pipe shall be used to convey streams containing β and γ sources. Then, it is assumed that the pipe is completely filled with an aqueous solution containing β and γ emitters homogeneously distributed.

For sake of simplicity, two different mono-energetic β and γ sources, having respectively energy equal to 174 keV and 661 keV, have been considered. These values correspond respectively to the average energy of the β particle and the characteristic energy of γ radiation emitted during the decay of Cs-137 that for its long half-life is assumed as the dominant radioactive contaminant that most likely could be contained in radioactive liquid waste streams in many industrial applications.

As a first step, the range of β particles in water has been assessed by means of the following empirical formula (L'Annunziata, 2003):

$$R_{max} = 0.11\left(\sqrt{1 + 22,4E^2} - 1\right)$$

where E (MeV) is the end-point energy β decay, and R_{max} (cm) is the range of β particles. With E equal to 0.512 MeV (end-point energy of

Cs-137 β decay), one gets

$R_{max} = 0.179$ cm

The result allows, for the simulation, to reduce the β source uniformly distributed in the entire pipe cross section to a source of β particles, located in proximity of the pipe inner wall. The penetration depth (D) of β particles in the bulk material has been computed by means of the following empirical formula (L'Annunziata, 2003):

$$D[\mathrm{g/cm^2}] = 0.412E_\beta^{1.265-0.0954\ \ln(E_\beta)}$$

where E_β is the average energy of β particles. For Cs-137, E_β=0.174 MeV; and considering for PVC an average density of 1.46 g/cm^3, one gets

$D = 0.2$ mm

Since the thickness of the PVC pipe considered for this application is 3.5 mm, the result shows that a large amount of the energy of β particles is expected to be deposited in the most inner layer of the pipe. Conversely, for γ radiation, due to the much higher penetration depth in matter, an almost uniform energy deposition inside the bulk material has been considered.

The simulation has been carried out by means of FLUKA code, a fully integrated particle physics Monte Carlo simulation package (Battistoni et al., 2007) developed jointly by CERN, the European Organization for Nuclear Research, and INFN, the Italian National Institute of Nuclear Physics. In order to assess profile of the energy deposited along the pipe thickness, the pipe (inner nominal diameter 10 cm; thickness 0.35 cm) has been modeled as a set of three adjacent discrete concentric circular shells. Number and thicknesses of the shells have been selected by taking into account the penetration depth of β particles computed with formula above reported.

Table 2 shows the dose (energy deposited per unit mass of bulk material) in each shell as a result of a single β and γ decay occurring in the radioactive solution.

The simulation confirms that the energy of the β particles located in proximity of the pipe is deposited only in the inner layer, while the energy of photons is distributed almost uniformly along the pipe thickness. As the doses reported in Table 2 are referred to a single decay, the table allows computing the dose on the pipe once the residence time of the aqueous solution and the concentration in Cs-137 are known.

2.2. Irradiation tests

Two separate β and γ irradiation tests have been carried out in air: for the β irradiation test, a LINAC, with a nominal power of 15 kW and generating a beam of high-energy (10 MeV) electrons, was used; γ irradiation tests were carried out with a device equipped with a Co-60 radioactive source. Both devices are conceived for the sterilization of medical products but the rate of release of dose to targets is very different: in fact, while it took approximately 8 h to provide a γ dose equal to 25 kGy, the same β dose required a few seconds application. For both tests, doses have been preliminarily selected based on information retrieved in literature, and showing that unplasticized PVC shows the first signs of degradation when dose exceeds some tenths kGy, while for higher doses deeper structure modifications are triggered (Campbell, 1981; Placek et al., 2003; Woods and Pikaev, 1993).

Specimens for irradiation tests have been obtained by die cutting from commercial pipes; five different specimens have been irradiated in air at each selected dose, in order to verify results reproducibility. For γ irradiation tests, 4 different dose values, namely, 50, 100, 250 and 500 kGy have been chosen, while β irradiation test have been carried out for a smaller set of dose values (50, 75, 100 kGy). Due to the different dose release rate of the two devices, therefore, γ irradiation tests required some weeks, while β irradiation tests were carried out in a much shorter time.

Table 2
Numerical simulation – β and γ doses (Gy) deposited in the pipe shells per single decay.

	Thickness (cm)	β Dose (Gy)	γ Dose (Gy)	Total dose (Gy)
Shell 1 (inner)	0.11	2.48 E-13	3.44 E-13	5.92 E-13
Shell 2 (central)	0.12	1.82 E-16	3.48 E-13	3.48 E-13
Shell 3 (outer)	0.12	1.42 E-16	3.18 E-13	3.18 E-13

2.3. Post-irradiation tests

Post Irradiation tests have been carried out in GDS-SIRCI laboratories and included observations of changes in the materials structure, tensile stress tests by means of an electronic dynamometer, fracture tests by means of a Charpy impact test machine. This last allows also the estimation of the energy absorbed by the specimen during the impact. Yield stress tests have been carried out in accordance with the method described in Technical Standard ISO 16422: 2006.

3. Results and discussion

3.1. Irradiation tests

Fig. 1 shows the macroscopic color change of the specimens at increasing γ doses: the specimen darkens and the color changes from white (non-irradiated specimen) to dark brown (specimen irradiated at the highest dose).

The well known effect for PVC comes from a dehydrohalogenation reaction resulting in a double bond along the chain backbone. Once a double bond is formed, the neighboring chlorine atom becomes an allylic site; allylic chlorine atoms are well known to be highly labile, and thus particularly susceptible to a subsequent abstraction reaction. The product (a stabilized, conjugated radical) can undergo a further reaction with another radical to yield two adjacent (conjugated) double bonds. The process can then repeat itself. The result is that PVC is particularly prone to forming species having extended conjugation (and thus absorption in the near UV and visible range) (Clough et al., 1995). Anyway, the susceptibility of polymers to radiation-induced discoloration has little or no

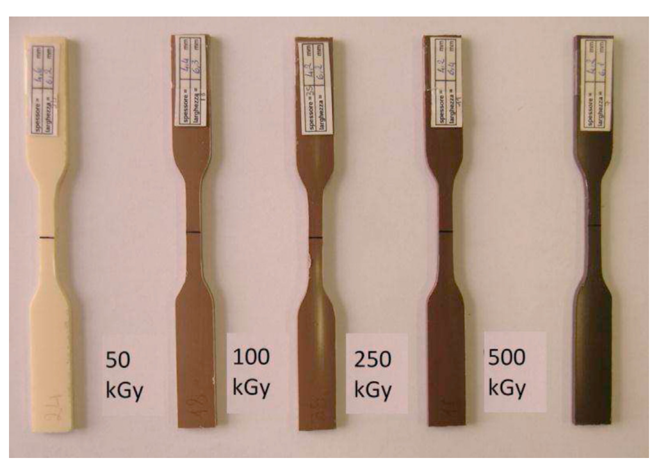

Fig. 1. Change of specimen surface color as effect of the dose (post γ irradiation test).

correlation with the susceptibility of these same materials to radiation-induced degradation in their mechanical properties (Clough et al., 1996). In PVC the permanent color centers are absorbed so strongly that it is not possible to see annealing even at the 'reverse' temperature.

Fig. 2 shows, for β and γ irradiated specimens, the trend of the yield stress, averaged over 5 tests, as a function of the dose.

For γ irradiated specimens the decrease in the yield stress begins at 50 kGy dose, although a sudden degradation of the mechanical properties, corresponding to a change in response of PVC to strain, occurs in correspondence of higher doses; in particular, at 250 kGy the yield stress sharply decreases; PVC is not anymore ductile but shows a typical fragile behavior; the specimen residual elongation at the rupture is lower than 50% of the elongation at the rupture measured for specimens non irradiated, or irradiated at lower doses.

The yield stress of specimens irradiated at a dose of 50 kGy is slightly lower (less than 5%) than the yield stress of non irradiated ones. The specimen residual elongation at the rupture is practically the same of non irradiated specimens, and corresponds approximately to 150% of the original specimen length.

Also in case of irradiation with β particles, results show that the yield stress in general decreases when radiation doses increase. At 50 kGy dose, a slight increase (1 MPa) in the measured yield stress has been observed; this increase is not significantly greater than the experimental errors of the tensile testing, so that the results do not allow to assess whether this can be due to the triggering of crosslinking phenomena. Radiation induced crosslinking in the material as a mean to enhance its mechanical properties, however, is beyond the scope of this work and therefore has not been investigated further; in fact, the experiments were basically aimed to assess the mechanical properties of bi-oriented PVC when exposed to given doses of ionizing radiation in order to ascertain whether this material as such could be considered suitable for nuclear industry applications.

Results of rupture tests are shown in Fig. 3. Up to a dose of 100 kGy specimens do not break or show incipient signs of fracture and absorb approximately 50% of the energy of the pendulum, thus showing the same behavior of non irradiated ones; at higher doses specimens show a fragile-type complete fracture, without significant energy absorption. Even if other irradiation tests performed on thin films of PVC with high drawings (Akay et al., 1980) shown an increased oxidative degradation, the tested Bi-oriented PVC still maintains a yield stress greater than 40 MPa up to 500 kGy; this suggests that, likely, its spatial orientation is not significantly altered by irradiation in a somehow.

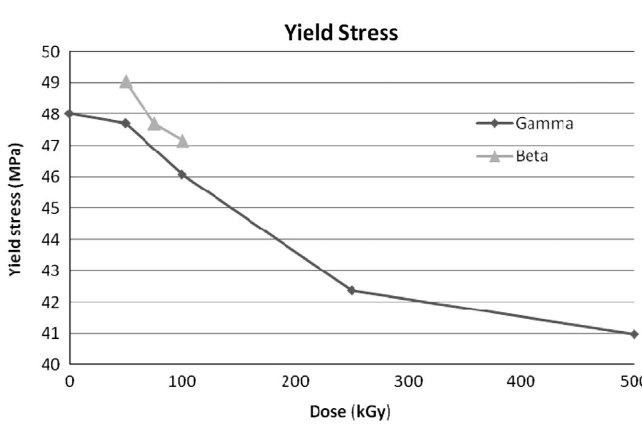

Fig. 2. Trend of the yield stress as a function of the dose (post β and γ irradiation tests).

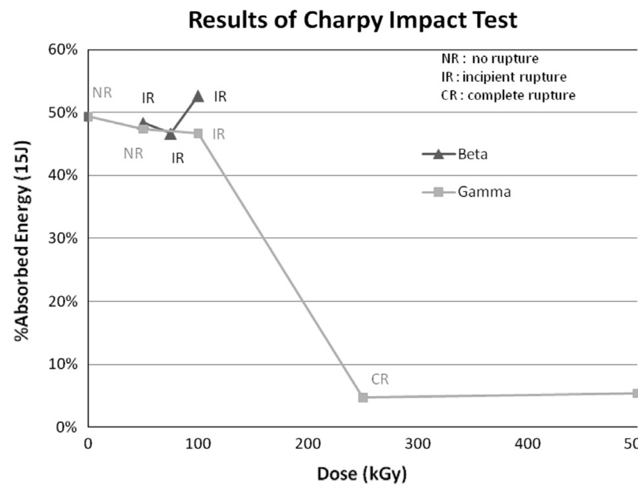

Fig. 3. Response to Chary Impact Test (post β and γ irradiation tests).

It has to be remarked that at the tested doses, although there is an increasing degradation of the mechanical properties when the doses increase, nevertheless the phenomenon of reverse never takes place; the shape and the dimensional characteristics of all the irradiated specimens remain unchanged, even for the highest doses. The spatial structure is lost only furnishing great quantities of heat up to 'reverse', this condition is obviously very far from the highest irradiations.

3.2. *Contamination and decontamination tests*

Simple contamination and decontamination tests have been carried out in order to preliminary assess the suitability of PVC in nuclear applications. For the tests an aqueous solution containing Cs-137, Sr-90, and Y-90 was used. The solution, with a specific activity of approx 3.8×10^7 Bq/l (Cs-137: 2.0×10^7 Bq/l; Sr-90: 1.8×10^7 Bq/l; Y-90: traces) is a nuclear waste deriving from the cleaning operations performed during the decommissioning of the research nuclear reactor L54M 'Enrico Fermi', formerly in operation at the Energy Department of Politecnico di Milano; for its characteristics the solution can be considered as representative of a liquid radioactive waste in many applications.

In the first (static) contamination test, a half pipe has been filled with an aqueous radioactive solution, in order to wet and contaminate the entire inner surface. After 90 h, the solution has been removed and the pipe section dried in air for 75 h. The second (dynamic) test aimed at the simulation of the effects of the flow of radioactive contaminants inside a process pipe. For this purpose a pipe, closed at both edges and partially filled with the solution, has been hold in continuous rotation at 60 rpm along its longitudinal axis for 140 h by means of an electrical motor. After, the radioactive aqueous solution was removed and the pipe dried in air (32 h drying time).

In both tests the radioactivity due to the deposition of radionuclides on the pipe inner wall was measured by means of a Geiger–Muller detector, at different pipe locations, in order to take into account the non uniform deposition of radioactive contaminants.

After the contamination the test pipes have been washed different times with fresh water and, after each washing operation, the residual activity has been measured in order to assess the effectiveness of the decontamination process. Fig. 4 shows the trend of radioactivity measured in a single pipe location in the static test as a function of the number of washing operations. Similar profiles have been obtained for both tests and for each pipe location selected for the measurement.

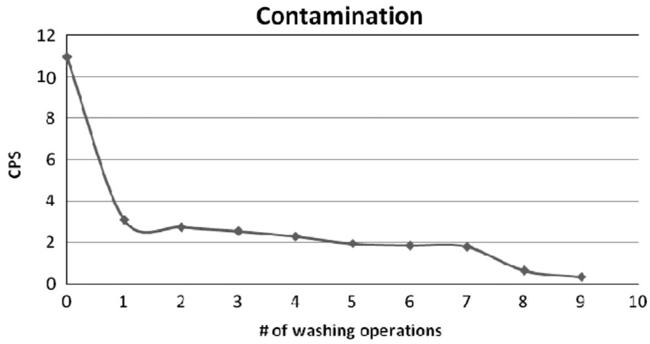

Fig. 4. Residual contamination (CPS) as a function of the number of washing operations.

Table 3
Average Decontamination Factors.

Test	No. of measurement locations	No. of wash. operations	Average DF	Std. Dev.
Static (half pipe)	5	9	31.60	3.93
Dynamic (rotating pipe)	4	2	16.18	0.86

As a final result, the Table 3 shows, for each test, the decontamination factor (DF), defined as the ratio between the radioactivity measured before the first washing operation and after the last washing operation performed. Values reported in the table are, for each test, averaged over all the pipe locations chosen for the measures.

The values of the decontamination factors achieved are in line with those expected after decontamination operations of metallic piping performed during decommissioning operations (Bregani, 1993).

3.3. Suitability of bi-oriented PVC for nuclear applications

Based on the experimental results, a preliminary assessment on the suitability of bi-oriented PVC in nuclear applications can be done. Both β and γ irradiation tests confirm that up to a dose of 100 kGy the mechanical properties of the material are practically unchanged, apart from an evident effect of change of the color. In fact, the yield stress decreases by less than 5% of the nominal value and the material remains ductile, as confirmed by Charpy impact test. Conversely, for higher doses there is a sharp degradation of the mechanical properties and the material shows a fragile behavior. However, for all the dose values that have been tested, the phenomenon of 'reverse', i.e., the loss of the orientation of polymer molecules in the bulk material does not take place. Should this phenomenon have occurred, the suitability of bi oriented PVC for nuclear applications would have been prevented a priori.

For a possible application of bi-oriented PVC in contact with radioactive fluids let us consider conservatively the total absorbed dose only in the first inner shell equal to 0.592 pGy/decay (Table 2). Such a dose rate requires 89.3 MBq to reach 50 kGy over a lifetime of 30 years. The simulations considered Cs-137 homogenously diluted in a water-equivalent liquid of 0.833 l, so, the maximum allowable concentration of radioactive contaminant in the pipe can be 107 MBq/l. The choice to limit the damage to the inner shell at 50 kGy protects the residual shells (the residual material in a cross section) allowing the tube to maintain its macroscopic properties.

This value is obtained by assuming the pipe has been for 30 years in continuous contact with that radioactive fluid and neglecting any kind of special corrosion effect due to the creation of vulnerabilities on the inner surface. Further investigations should be undertaken considering the possible formation of craters on the inner surface deriving from oxidative species digging into tracks created by β particles.

A radioactive stream with a higher specific activity could be safely managed for a shorter time period, or conversely, in case of intermittent/discontinuous flow (as for the case of potentially contaminated waste water) the pipe lifetime could be significantly extended. The estimated lifetime, in combination with the maximum allowable concentration of radio-nuclides in the conveyed streams, the good resistance to chemical corrosion and the preliminary results of decontamination tests in view of future decommissioning operations, suggest that the material is suitable for different nuclear applications, including waste deposits for low and intermediate level nuclear wastes, as well as for radioactive waste recovery systems for nuclear medicine applications.

4. Conclusion

An assessment of suitability of bi-oriented PVC pipes in nuclear applications, as a possible replacement of metallic piping, has been done. Due to the orientation of polymer chains along preferential directions, result of the manufacturing process, bi-oriented PVC shows enhanced mechanical properties with respect to traditional, unplasticized PVC. Specimens cut from bi-oriented PVC commercial pipes have been irradiated with γ rays and β particles. For γ irradiation tests the doses ranged between 50 and 500 kGy, while for β irradiation tests the doses ranged between 25 and 100 kGy. Dose values were selected on the basis of data reported in literature, and showing that in correspondence of some tenths kGy PVC shows first signs of degradation.

For γ irradiated specimens, the decrease in the yield stress begins at 50 kGy dose, although a sudden degradation of the mechanical properties, corresponding to a change in response of PVC to strain, occurs in correspondence of higher doses (250 kGy). Also in case of irradiation with β particles, results show that the yield stress in general decreases when radiation doses increase.

Rupture tests show that up to 100 kGy dose specimens remain ductile, do not break or show incipient signs of fracture behaving like non irradiated ones; at higher doses specimens show a fragile-type complete fracture. However, for all the doses that have been tested, the phenomenon of 'reverse', i.e., the loss of the orientation of polymer molecules in the bulk material, turning into a macroscopic change of size and shape in the pipe and into the complete loss of the enhanced mechanical properties due to the bi-orientation process, does not take place.

Simple contamination tests have been carried out, by filling pipe samples with an aqueous solution containing radioactive contaminants. The solution, containing β and γ emitters, is a waste deriving from the decommissioning operations of a nuclear reactor used for research, and can be considered as a representative of typical radioactive waste liquid streams in nuclear applications. After the deposition of solid contaminants, the pipe samples have been cleaned, and the decontamination factor assessed. Results show that the decontamination of PVC pipes does not pose significant problems with respect to other materials.

In order to compare the effects of radiation expected on the pipe in nuclear industry applications with those resulting from the irradiation tests, a numerical simulation has been carried out. Assuming conservatively 50 kGy as the maximum allowable dose that the pipe can withstand without significant damage, it turns out that for a reference stream conveyed by the pipe and containing Cs-137 with a specific activity slightly less than 100 MBq/l, the expected pipe lifetime is approximately 30 years. This result, in combination with the enhanced mechanical properties, the good resistance to corrosion and the preliminary results of decontamination suggest that bi-oriented PVC could be suitable for nuclear applications, including waste deposits for low and intermediate level nuclear wastes, as well as for radioactive waste recovery systems for nuclear medicine applications.

Acknowledgments

The authors wish to thank Prof. M. Mariani and V. Varoli (Centro Studi Nucleari Enrico Fermi, Politecnico di Milano) for their contribution in the experimental tests and the interpretation of the results, and A. Capponi (Tecnimont S.p.A.), I. Tripputi (Sogin – Società Gestione Impianti Nucleari) and F. De Angelis (Ansaldo Nucleare) for their contribution in the discussions about the suitability of PVC in industrial and nuclear applications.

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