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Experimental Validation of a Test Apparatus for the Evaluation of Hydrogen Permeation in Silane-Modified Sealants on Fuel-Cell-Powered Vehicles

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Abstract: Silane-modified sealants are widely used for the construction of railway vehicles and have several advantages in the production of elastic structural joints and seals featuring high bond thickness. The use of hydrogen fuel cells to power newly developed rolling stock places further safety constraints on the design of the sealing elements of those technical compartments that contain the storage tanks of the propulsion system. Given the lack of solutions based on the use of silane-modified sealants validated for operating environments in which leaks of gaseous hydrogen may occur, an experimental test was carried out to characterize the permeability of some adhesive products according to the requirements of the BS ISO 15105-2:2003 standard, and a specific test bench was developed for this. Two different sealants were subjected to the hydrogen permeability test. The processing of the results provided by the apparatus designed specifically for the execution of the test made it possible to determine a permeability rate dependent on the thickness of the adhesive in the order of ng/(min × cm^2). The results of the test were subsequently contextualized within the technical application to rolling stock, with the ultimate aim of verifying that the permeability rate determined experimentally is compatible with the design safety criteria. The developed test bench allowed the correct execution of the permeability test. In general, the two sealants showed hydrogen permeability values compatible with the application. In particular, the hydrogen permeation rate (Rp) was lower than 0.25 ng/min for both sealants.

Keywords: hydrogen; permeability; sealant; transport; railway

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1. Introduction

Nowadays, one of the most important challenges is the reduction of greenhouse gas emissions and pollutants deriving from human activities. In particular, the transportation sector produces about 25% of total emissions in the European Union [1]. It is therefore clear that reducing emissions in the transport sector can contribute significantly to reducing overall pollution. For rail transport, there are two types of means used in the EU:

- Electric traction, the realization of which is justified in economic terms by the presence of high traffic flows;
- Diesel traction, which can produce high emissions that are harmful to health.

The diesel traction for rail transport represents approximately 50% of the EU railway network [2]. Diesel trains increase pollution that can cause health consequences, especially in the most populated urban areas. For this reason, there are numerous alternative solutions being studied, such as for the automotive field, in order to improve the environmental impact of the European railway network. An example of these technologies is the use of biodiesel for rail transport. However, this feeding system has both benefits and problems.

In particular, the use of biodiesel has the health benefits of reducing the emissions of substances harmful to health, but it involves higher costs for fuel and unknown factors related to the adaptation of existing locomotives and infrastructures [3]. Another interesting technology that can be considered is fuel cells (FCs). FCs are electrochemical devices capable of producing electrical energy directly from some substances, such as hydrogen and oxygen, without any type of thermal combustion. In this way, there are no emissions of substances harmful to health, only water vapor. Hydrogen is a clean energy fuel when it is produced from renewable sources of energy [4]. In recent years, many transport projects have focused on the use of hydrogen in vehicles [5–7]. For this reason, focusing on the use of hydrogen in vehicles is growing. For example, the HydroFLEX project was developed in the UK in October 2018. This project combined many different engineering disciplines to achieve a singular objective—to make an electric train self-powered by adding a hydrogen-hybrid power system. The HydroFLEX team showed the possibility of operating hydrogen-hybrid trains on the British railway network [8]. However, the possible use of hydrogen-hybrid trains would certainly require the railway infrastructure to be adapted for these means [9]. On the other hand, a hydrogen train can reduce energy consumption by up to 34% compared to a diesel–electric train [10]. Charging stations for hydrogen trains are also a sustainable investment, both economically and environmentally. The investment has an 85.6% probability of being successful with the appropriate measures [11]. However, it is important to convince the population of the need for energy conversion in transport, including through economic incentives from governments [12]. For this reason, continuous applied research in this area is mandatory. For example, Abaza et al. proposed a novel swarm-based algorithm called the coyote optimization algorithm (COA) for the parameter optimization of PEM fuel cells as well as PEM stacks, with significant accuracy of the proposed method [13]. Moreover, Abo-Elyousr et al. developed two probabilistic self-adjusted modified particle swarm optimization (SAPSO) algorithms. The two algorithms were compared to minimize total operational costs, addressing all constraints of the distribution system, DG (distributed generation) units, and energy storage systems of EV (electric vehicle) parking lots [14]. Furthermore, Zhou et al. developed a finite element model to investigate the sealing performance of the combined seal structure constructed of a rubber O-ring and a wedge-ring under ultrahigh pressure gaseous hydrogen [15].

The use of hydrogen trains also entails the need to carefully evaluate the related safety issues, considering that hydrogen is a flammable and explosive gas. Elements particularly exposed to possible gas leaks are those sealing joints where polymeric adhesives are often used. In particular, adhesive systems modified using polyether-based silane compounds (silane-modified polymers) are widely used in the railway transport sector for the construction of a wide range of rolling stock. Silane-modified polymers are mainly single-component adhesives characterized by an organic structure based on ether functional groups coupled with inorganic silane groups. The crosslinking process of the adhesive in the resin state consists of multi-stage polycondensation triggered by the humidity present in the air. During crosslinking, alcohols are released as reaction compounds, and the resulting product has a typically elastomeric structure in which oxygen atoms are present and act as a bridge between the polymer chains. Precisely, this characteristic gives silane-modified adhesives good mechanical strength and excellent flexibility, with typical values of percentage elongation up to 800%. Other favorable features of silane-modified adhesives are:

- good adhesion on a wide range of substrates of different nature;
- possibility of use over a wide temperature range, indicatively from −40 to 100 °C;
- high resistance to UV (ultraviolet) rays.

Nowadays, the design of adhesively bonded joints with silane-modified sealants systems must take into account new types of requirements, in addition to those already mentioned, dictated by the increasingly urgent demands of transport sustainability and ecological protection. The hydrogen fuel cell can represent a valid solution for the production of energy to power railway vehicles, where electrified infrastructures are absent.

However, this hydrogen-based technology poses new challenges in terms of sealing that can be guaranteed by the adhesives used as sealants. In November 2020, the board of directors of Ferrovie Nord Milano (FNM), the main Lombard public transport group, commissioned Alstom, an important French rolling stock manufacturer, to supply six regional trains powered by hydrogen fuel cells. These new trains are based on Alstom's Coradia Stream regional platform and will be equipped with the same fuel cell propulsion technology of Coradia iLint, the world's first hydrogen-powered passenger train, which has already been in commercial service in Germany since 2018. The trainset's propulsion system is installed on a single vehicle called the power car (PwC). In addition to fuel cells, the PwC vehicle also carries hydrogen storage tanks that are installed inside two technical compartments arranged along the sides. The PwC vehicle is not intended to carry passengers; however, it has a central corridor that can be traversed to transit from one end of the train to the other. For reasons of industrial convenience, the walls that separate the central corridor from the lateral technical compartments are made of sheet metal covers screwed to the primary structure of the PwC. This feature makes it necessary to check the safety of the vehicle in the remote event that hydrogen gas diffuses into the technical compartments due to unnoticed leaks in the storage system. In particular, the adequacy of the seal to be applied along the entire perimeter of each sheet metal cover must be assessed. It is important to point out that in the railway normative panorama, there is currently no complete regulatory plan regarding hydrogen-propelled trains. For safety issues, reference is made to standards that, while related to hydrogen propulsion, are applicable to other sectors. However, these standards can only be partially transposed into the context of rolling stock, so risk mitigation strategies often follow the path of engineering common sense and rely on the support provided by the on-field return of experience. For silane-modified adhesives, hydrogen permeation is certainly a relevant issue, considering the fact that the microstructure of these materials already allows for a migration of the products resulting from the polycondensation process during the crosslinking phase. It was, therefore, defined as the objective of the safety check to quantify the time required to reach a concentration of 4% within the corridor of the PwC due to the passage of gaseous hydrogen through the sealing seams following a failure of the storage system, leading to the propagation of fuel inside the technical compartment. The 4% threshold represents the lower limit of the flammability range of hydrogen mixed with air in SATP (standard temperature and pressure) conditions (25 °C and 1 bar). The result of the verification must then be compared to the timescales that are typical of the different operational situations of the trainset. In the absence of explicit normative references, we decided to proceed by adopting an empirical–experimental approach. In this analysis, it was taken into account that leaks of gaseous hydrogen H_2 through the sealant beads can occur in two different ways:

- primary leaks through interstices between the adhesive and the substrates;
- secondary leaks due to hydrogen permeation and diffusion phenomena through the adhesive.

Primary leaks can be avoided by qualifying a robust bonding process that is able to ensure proper adhesion between the adhesive and the substrates. Adhesion problems can also be avoided with appropriate design choices, focusing on the geometry of the joint, the aspects related to corrosion that could arise at the interface, and the effects of adhesive aging. As regards secondary leaks, the gaseous permeation of a polymer is a complex physicochemical process that takes place in different phases at the two ends of the solid medium: solution and absorption on the side of the surface exposed to the gas, and diffusion inside the medium and release by desorption on the opposite side.

The safety check performed for this purpose was divided into a series of experimental tests with the aim of defining the hydrogen permeability rate of the adhesives that can be used in the design of the sheet metal roofing present on the walls of the PwC corridor. There are multiple techniques for characterization tests of the permeability properties of polymeric materials. Among the main ones include gravimetry, techniques that use the magnetic suspension balance, and others based on manometry. The scientific literature also provides several recent research projects that have used gas chromatography as a characterization technique [16–18].

The methodology described in this paper focuses on determining the specific properties of hydrogen permeation in sealants through the setup of a suitable test system in order to guide the subsequent design phase of the joining application. For this reason, the test bench was designed by BeonD, an Italian engineering company active in the field of innovation and "green" applications. The following are the advantages of the bench:

- It reduces the hydrogen leaks to evaluate the hydrogen permeability of the tested materials correctly;
- It allows the testing of sealants of different thickness (1–6 mm).

2. Materials and Methods

According to BS ISO 15105-2:2003 [19], a permeability test was carried out to evaluate the hydrogen permeability of two polymeric sealants produced by Vaber Industriale S.p.a. (Torino, Italy), an Italian company that produces adhesives for industrial use in the transportation fields (automotive, railway, etc.):

- Advantseal FR (fire retardant), a single-component sealant suitable for sealing joints and metal overlaps, with intumescent properties that make it suitable for the construction of fire barriers [20];
- Cristalcar Plus, a single-component adhesive suitable for semi-structural elastic bonding, particularly suitable for the assembly of glass or windshields [21].

A customized test bench was designed by BeonD for the hydrogen permeability test. In Figure 1 and Table 1, a diagram of the test bench and its legend is reported. A nitro hydrogen mixture (95% N, 5% H_2) was selected as a test gas instead of a dry air mixture to ensure a sufficient concentration of hydrogen for safety purposes since, at concentrations higher than 4% in the air, hydrogen can become explosive.

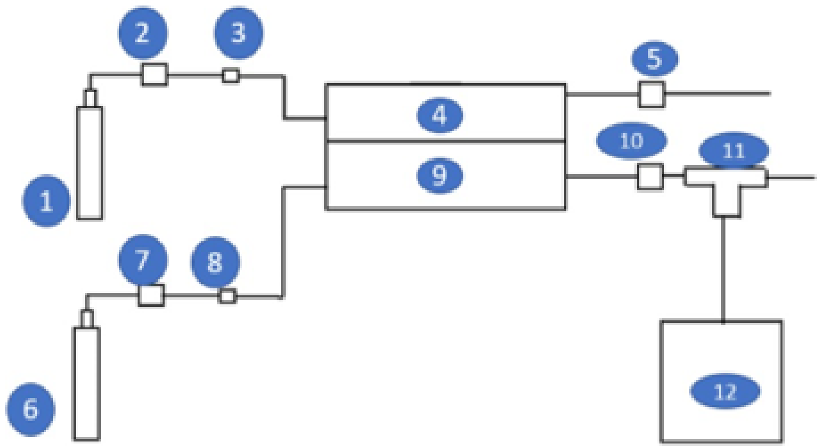


Figure 1. Diagram of the hydrogen permeability test bench.

The principle of operation of the test is based on the use of a permeable cell, inside which the adhesive test sample is mounted as a separator septum between two sealed chambers (4 and 9). The test chamber was designed by Beyond and is shown in Figure 2. The upper chamber is fed with the test gas; the lower chamber is lapped by a regular flow of gas called a conveyor. The total pressure is the same in each of the two chambers, but the test gas is at a higher partial pressure in the chamber it occupies. For this reason, the test gas permeates the sample and migrates inside the chamber where the carrier gas flows. The gas molecules that permeate the adhesive septum are conveyed by the carrier gas to a continuous cycle sampler, which repeatedly injects its contents into a gas chromatograph.

Table 1. Legend of hydrogen permeability test bench.

Number	Component
1	Nitro hydrogen cylinder
2	Nitro hydrogen adapter
3	Nitro hydrogen needle tap
4	Upper chamber
5	Nitro hydrogen flow meter
6	Argon cylinder
7	Argon adapter
8	Argon needle tap
9	Lower chamber
10	Argon flow meter
11	"T" fitting
12	Gas chromatograph

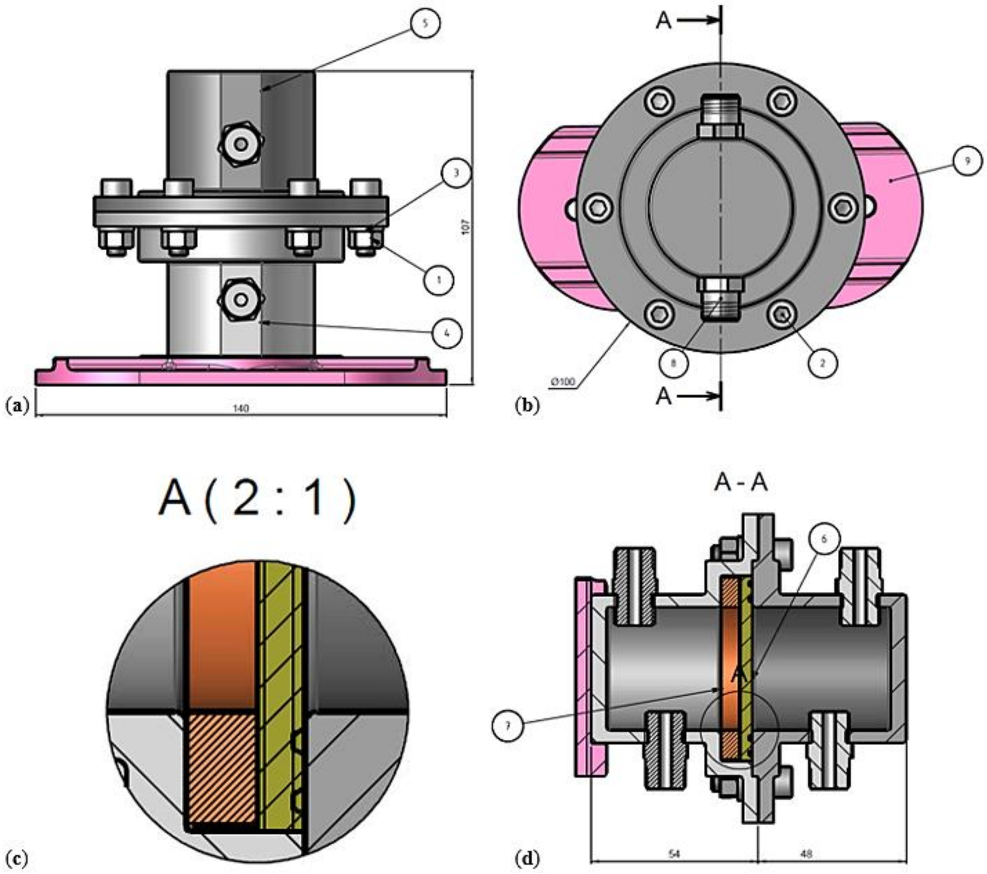
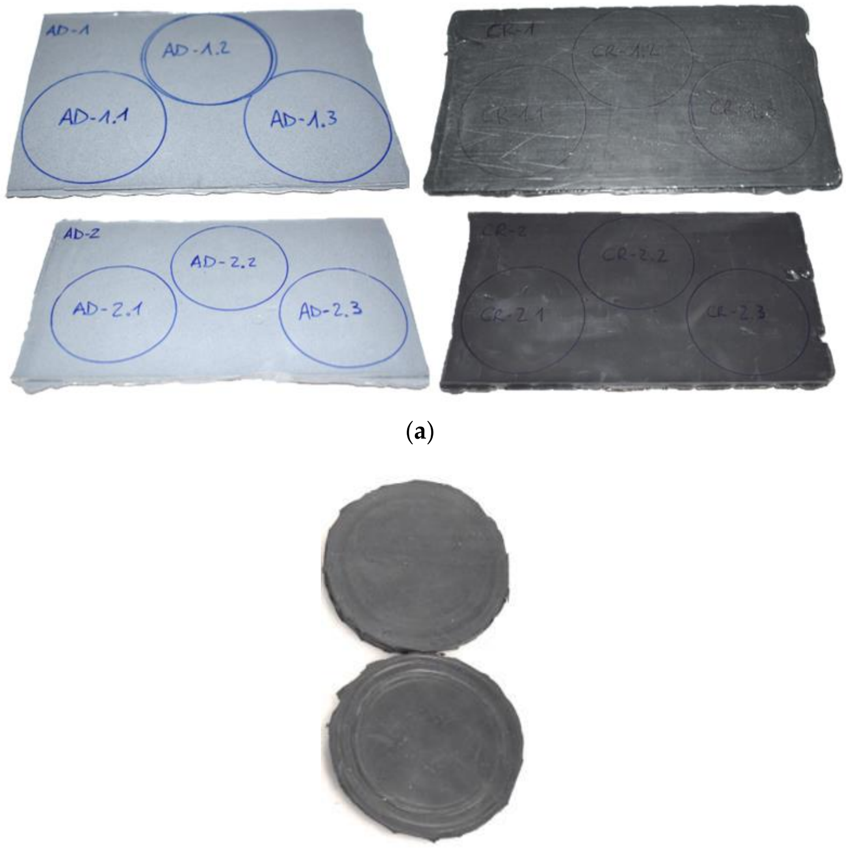


Figure 2. Design of hydrogen permeability test chamber. (**a**) Front view; (**b**) top view; (**c**) position of the specimen; (**d**) lateral section.

A TCD (thermal conductivity detector)-type detector is associated with the gas chromatograph. The detector records the changes in thermal conductivity of the gas from the chromatographic column and compares it to a reference flow of the carrier gas. In this way, all compounds that have a different thermal conductivity than the carrier gas are detected. The reducers, located downstream of the supply cylinder delivery, are used to regulate the pressure of the gaseous flows in such a way as to achieve the condition of equal pressure between the two chambers separated from the sample. The test samples are discs of pure adhesive, with a diameter of 60 mm, obtained from rectangular sheets of homogeneous thickness by crosslinking the materials subject to the test. The rectangular sheets were prepared and supplied by Vaber according to the indications specified on the TDS (technical datasheet) of the adhesives (environmental conditions, crosslinking times, etc.). In Figure 3, the rectangular sheets and the samples used for the hydrogen permeability test are reported.



(**a**)

(**b**)

Figure 3. (**a**) Sealant rectangular sheets. (**b**) Specimens for hydrogen permeability test.

The test chamber consists of two cylindrical flanged chambers that are assembled by interposing the adhesive sample disc between them, which also acts as a sealing element. The addition of a spacer ring makes it possible to ensure the tightness of the chambers by compensating for the differences in the thickness of the various samples. The arrangement of the various elements favors the permeation of the samples in a direction perpendicular to the faces of the discs, making less likely the occurrence of diffusive phenomena oriented towards their radial ends. In Figure 4, the assembly of the test apparatus is reported.

The test apparatus is equipped with a monitoring system of environmental parameters and hydrogen levels with redundant signal acquisition. In particular, the temperature of the cell chambers is detected by means of type K thermocouples applied to the external cylindrical walls. The flow of gases leaving the two test chambers is also monitored by means of dedicated flow meters in order to correctly determine the hydrogen permeability rate without the incidence of altering effects due to leaks at the fittings.

The qualitative and quantitative analysis of the chemical species transported by the gas leaving the test cell was performed by an Agilent 490 gas chromatograph provided by Professor Claudio Minero of the Chemistry Department of the University of Turin (UniTO). Each measurement consisted of 60 min of continuous sampling for a total of about 10 gas-chromatographic analyses per measure and 4 measures per specimen in order to obtain regular stabilization of the system for each measure. Knowing the flow through the lower chamber, the hydrogen permeation rate (R_p) in ng/min through the membrane was determined by Equation (1) [17]:

$$R_p = \frac{CFM}{24.45} \quad (1)$$

where C is the hydrogen concentration in ppmv, F is the flow rate in mL/min, M is the molar mass of hydrogen gas (2.016 g/mol), and 24.45 is the molar volume of hydrogen (L/mol). The specific permeability rate (R_{ps}) in ng/(min × cm^2) was also calculated using Equation (2) [17]:

$$R_{ps} = \frac{R_p}{A} \tag{2}$$

where A is the specimens' specific area in cm^2. In Figure 5, the entire test bench is shown.

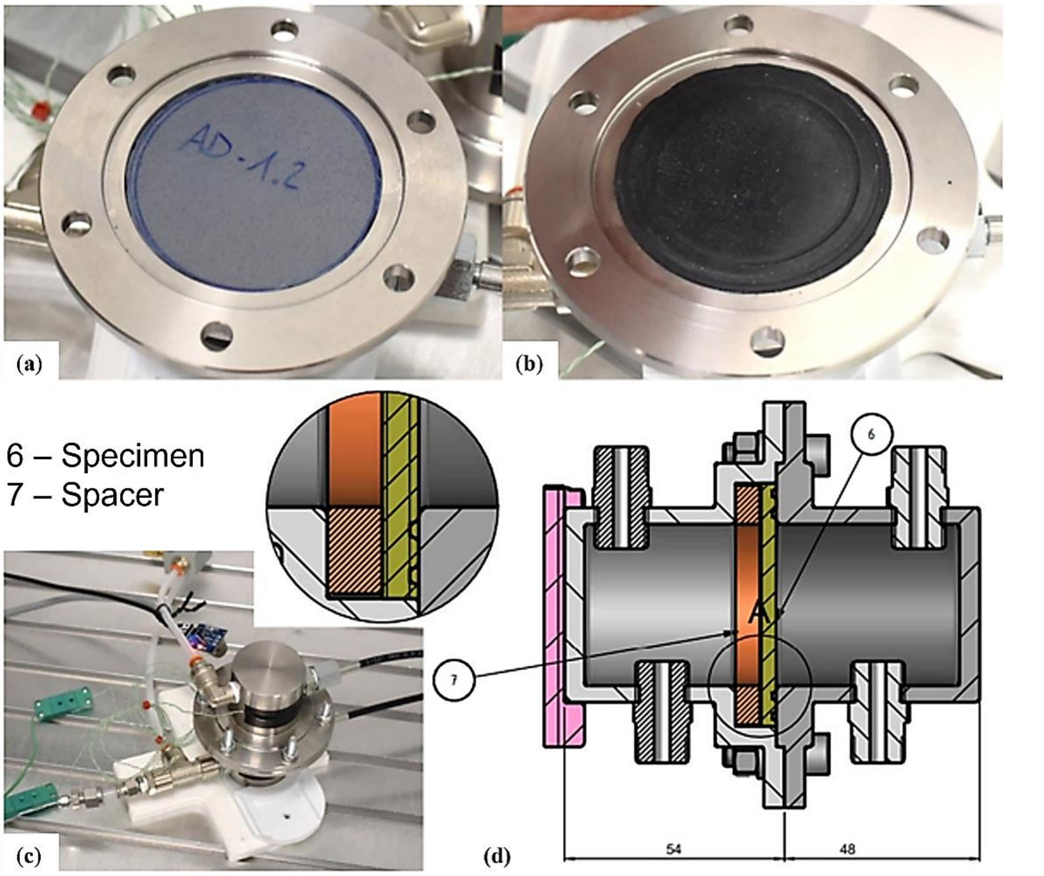


Figure 4. Assembly of the hydrogen permeability test apparatus. (**a**) Advantseal specimen. (**b**) Cristalcar specimen. (**c**) Test chamber with hydraulic connections. (**d**) Test chamber's lateral section.

Figure 5. Permeability hydrogen test bench.

3. Results and Discussion

In Table 2, the results of the hydrogen permeability test are reported. The sealant Advantseal (AD) FR has a lower specific permeability rate than the Cristalcar (CR) Plus sealant. However, both 6 mm thick sealants have permeability values lower than the sensitivity of the gas chromatograph; at 3 mm thick, the permeability values are, in any case, lower than 3 ng/min.

Table 2. Experimental results hydrogen permeability test.

9	Measure	Nominal Thickness [mm]	R_p [ng/min]	R_{ps} [ng/(min*cm^2)]
Advantseal FR	1	3	1.9	0.15
	2		2.2	0.18
	3		2.3	0.18
	4		2.1	0.17
	1	6	<0.6	<0.05
	2		<0.6	<0.05
	3		<0.6	<0.05
	4		<0.6	<0.05
Cristalcar Plus	1	3	2.6	0.20
	2		2.6	0.21
	3		2.3	0.19
	4		2.8	0.23
	1	6	<0.6	<0.05
	2		<0.6	<0.05
	3		<0.6	<0.05
	4		<0.6	<0.05

In Figure 6, a chromatogram of the permeability test results is reported. The graph has been offset by a fixed amount to allow the best visualization and reading, as is usually the case in chromatographic studies. The parameter to be taken into consideration when reading the graph is the area subtended value of the peak with respect to the baseline of its curve. The time indicated on the *X*-axis starts from the instant in which the gaseous species enter the column of the gas chromatograph. The release time of the molecules of the chromatographic column depends on the properties of the column itself and is always the same for the same chemical species. For 6 mm thick sealants, there is no significant peak in correspondence with hydrogen. The peaks of oxygen and nitrogen recorded are attributed to a small permeation of air through the PA6 plastic pipes of the system, negligible for the purposes of the test since it is not able to vary the quantity of hydrogen permeation. For 3 mm thick sealants, significant H_2 peaks for both sealants were registered. The Cristalcar hydrogen peak is greater than the Advantseal hydrogen peak, in accordance with the permeability values indicated in Table 2. The hydrogen sensors constantly monitored the amount of ambient hydrogen, which was always not significant. Therefore, it can be said that:

- For 6 mm thick sealants, there is no hydrogen permeation detectable by the gas chromatograph, resulting in less than 0.6 ng/min (instrument sensitivity);
- For 3 mm thick sealants, there is hydrogen permeation below 3 ng/min, higher for Cristalcar than for Advantseal.

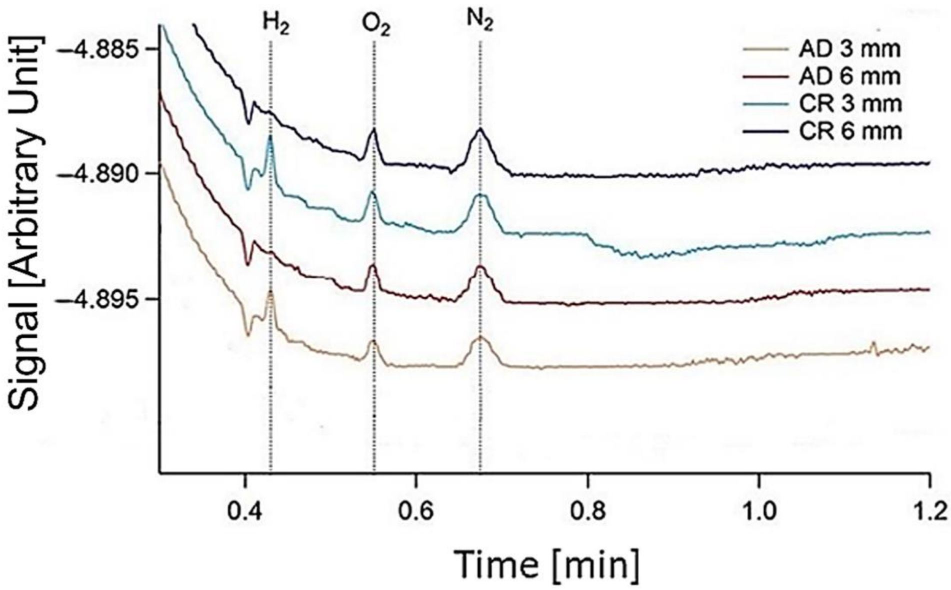


Figure 6. Permeability test chromatogram.

4. Conclusions

In the present work, the hydrogen permeability of two industrial polymeric sealants produced by Vaber, Advantseal FR and Cristalcar Plus, was evaluated. This analysis was used to carry out design checks for the PwC vehicle of the "Coradia Stream" train for FNM, developed by Alstom. In particular, it was possible to determine the safety timescale in case of hydrogen leaks on board the train itself. In Table 3, the hydrogen permeability average results are reported. The results for both the tested sealants revealed negligible hydrogen permeation (less than 0.25 ng/(min $\times$ cm^2)) for 3 mm thick sealants and hydrogen permeability not detectable by the gas chromatograph for 6 mm thick sealants, which allows them to be used safely even in the event of significant hydrogen leaks on board the train. In particular, the Advantseal hydrogen permeability is lower than Cristalcar. The result is absolutely acceptable even in relation to the operating situation in which the train can remain stationary without power for relatively long periods. The test apparatus set up by BeonD, validated on this test cycle, can be used for conducting new experiments aimed at investigating other relevant aspects, such as:

- Permeation properties of adhesives following aging cycles;
- Influence of the variation of environmental conditions (temperature, humidity, etc.) according to the minimum and maximum limits set by the train mission profile;
- Evaluations on the possible use of sealants for pressure applications, which are more interesting in the high-speed field (in this case, the regulatory reference for the test methods would be Part 1 of the BS ISO 15105 standard).

Table 3. Hydrogen permeability test average results.

Material	Specimen	Nominal Thickness [mm]	R_p [ng/min]	R_{ps} [ng/(min$\times$cm^2)]
Advantseal FR	AD	3	2.1 ± 0.1	0.17 ± 0.01
		6	<0.6	<0.05
Cristalcar Plus	CR	3	2.6 ± 0.2	0.21 ± 0.02
		6	<0.6	<0.05

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