CH_4 Permeability through PE Liners – Testing Report

Experiments carried out by:	**Mecadi GmbH** Industriegebiet In der Kolling In der Kolling 9 66450 Bexbach (Germany) Phone: +49-(0)68 26-933 83 0 Fax: +49-(0)68 26-933 83 120 Email: info@mecadi.com Internet: www.mecadi.com
Customer	Plastika Kritis S.A. P' STR. INDUSTRIAL AREA GR 714 08 Iraklion, Crete
Purchase Date	18.09.2015
PO Number (Customer)	-
Test Sample Sample N°	PE liner see table
Project N° Mecadi	1005
Vendor N°	-
Testing Period	02.10.2015 – 22.10.2015
Total Page Number	5

Methodology

Gas Permeation via pressure increase method:

- Experiment in accordance to ISO 15105 (pressure increase)
- Apparatus: Mecadi01302
- Permeation relevant area: 23.76 cm²
- Pressure transducer at the feed side: WIKA D-10-P (range: 0-10 bar) and (range: 0-100 bar)
- Pressure transducer at the permeate side: WIKA D-10-P (range: 0-1.6 bar)
- Testing temperature: 20.0 °C. Test temperature is maintained constant over the entire testing period employing a laboratory thermostat
- Relative humidity: 0 %RH
- Feed pressure: in the range of 4-5 bar absolute
- Permeate pressure: 25 - 100 mbar
- Testing gas: CH_4

Samples

Table 1: Listing of the employed samples

Sample N°	Average Thickness / mm	Testing gas
1005-151007-001	0,420	CH_4
1005-151012-001	0,424	CH_4
1005-151015-001	0,427	CH_4

Sample Preparation

Three large PE liner foils are delivered from the customer. From each piece of foil one test sample is cut with the approximate dimensions 10x10 cm². The so-obtained sample is then employed without any other preconditioning for the permeability measurements.

Sample Thickness

The sample's thickness is obtained by arithmetic averaging of 10 independently measured points utilizing the mechanical sensing technique (in accordance to DIN 53370).

Experimental

Pressure Increase Experiments – CH_4 Permeability

The sample to be tested is clamped in-between the two sides of the permeation measurement cell. During thermosetting each bold is screwed by hand. After the set temperature is reached a torque amounting preferably in-between 5-15 Nm is applied.
The experimental determination of the gas permeability using the pressure increase technique is divided into four steps. Before each measurement, the lateral as well as the overall tightness of the sample and the apparatus are verified.

• Degassing:
Process step to remove volatile components from the polymeric sample. Typically, degassing in done within 20-30 h depending on the polymer class, the material thickness and the degassing tendency. The preferably employed pressure amounts to 1-6 mbar. The degassing temperature is set to the later test temperature.

• Individual-Zero:
The time-dependent pressure increase within the per-evacuated permeate side, due to sample and/or cell specific leakage is called individual zero. The therefore employed time period is preferably chosen in the same range than the later pressure increase measurement time. The so-obtained leakage value is subtracted from pressure increase rate obtained during the pressure increase measurement.

• Pressure increase experiment:
Before measurement both cell sides are evacuated. At the starting point the feed side is pressurized with a test gas partial pressure of 4-5 bar (absolute). During the measurement the pressure increase in the permeate side due to the penetrant permeation is detected and logged. From the obtained pressure increase curve the diffusion coefficient is calculated from the early transient region of the curve, whereas the permeation coefficient is obtained from the quasi-stationary region.

• Volume permeate side
In order to determine the exact volume of the permeate side, a sample loop with known volume is connected to the measurement cell. From the change in pressure, observed when the valve in-between sample loop evacuated permeate side is opened, and the atmospheric pressure the exact volume of the permeate side is determined.

Results and Discussion

Table 3: CH_4 permeation data of the investigated PE liner samples at 20 °C.

Material	Sample N°	p_{feed} (CH_4) / atm	P-rate / cm³(STP)·m^{-2}·d^{-1}·atm^{-1}	T / °C	Signal to Zero ratio
PE liner	1005-151007-001	5.6	**< LOD**	20.1	1
PE liner	1005-151012-001	4.7	**< LOD**	20.0	1
PE liner	1005-151015-001	5.0	**< LOD**	20.0	2

STP Standard temperature and pressure, refers to 273.15 K and 1.013 bar. LOD = Limit of Detection = 12 cm³(STP)·m^{-2}·d^{-1}·atm^{-1}

Please note that the here reported values for the permeation rate are characterized as below lower limit of detection since the found signal to zero ratios are below 5 (see Table 3).
Basing on the carried out experiments an mean limiting methane permeation coefficient amounting for < 12 cm³(STP)·m^{-2}·d^{-1}·atm^{-1} at 20 °C is certified.
Please note this value is only valid for the tested samples. Any change in polymer micro and/or macro morphology (thermal, chemical, mechanical, etc. degradation) can significantly influence the permeability.

Project Team

Position	Name	Qualification
Projectmanager	Dr. Andreas Konrad	Chemist
Lab	Francesco Arena	Dipl.-Chem.
Lab	Wolfgang Molter	Quality Manager
Data treatment, Report	Francesco Arena	Dipl.-Chem.

Responsibility for Report Generation

	Name	Qualification
Report enabled: 23.10.2015	Dr. Andreas Konrad	Chemist
Report reviewed: 23.10.2015	Wolfgang Molter	Quality Manager
Report created: 23.10.2015	Francesco Arena	Dipl.-Chem.