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IVV

Résumé of the test report

Permeation testing of flexible packaging films with respect to mineral oil components

This is an excerpted version of the original test report issued by the Fraunhofer Institute for Process Engineering and Packaging on October 22, 2012. It may be used as a whole without restrictions in contact with third parties. The results relate only to the investigated samples.

1 Scope

The purpose of this résumé is to show the different behaviour of four selected films with respect to the permeation of selected mineral oil components. The samples were tested with respect to their potential to act as a functional barrier against mineral oil components under typical conditions for food-packaging contact and compliance tests (40 °C).

Whereas the full report as supplied on October 22, 2012 had the purpose to give an overview on a larger number of samples with respect to their behaviour upon permeation of many different substances, this report concentrates on four selected films, namely BOPP ("sample A"), a barrier film on the basis of PLA (Nativia NTSS 25 ,"sample C"), two multilayer films from conventional polymers, one with an EVOH barrier layer (EXTENDO XFWL 20, "sample D"), the other coated by PVDC and an acrylic layer ("sample K"), together with a PET film as a reference. For clarity of the presentation, a further selection among the permeated substances was done vis-à-vis the original report. This selection, however, still gives a representative comparison of the four selected films with respect to their permeability for mineral oil components.

It has to be said that, at the present state of the art, only the permeation rates of the samples for the selected substances can be given. The indication of a single value of a lag time, as presently done by several other researchers, is highly questionable. Also in the future it will only be possible to indicate lag times for single

permeating substances – not for mixtures of different substances - , but even this requires more precise information than presently available.

2 Test substances

The following list of substances was agreed as relevant representative substances for mineral oil, which, at the same time, allow for a good quantification of the permeation rates under stationary conditions.

Substance	CAS-Nr.	Formula	mw	mp	bp	density
			(g/mol)	(°C)	(°C)	(g/ml)
Dodecane (C12)	112-40-3	$C_{12}H_{26}$	170,33	-9,6	216	0,7487
Naphthalene	91-20-3	$C_{10}H_8$	128,17	80	218	1,1400
1-Methylnaphthalene	90-12-0	$C_{11}H_{10}$	142,20	-31	245	1,0200
Tetradecane (C14)	629-59-4	$C_{14}H_{30}$	198,39	6	254	0,7628
1-Ethylnaphthalene	1127-76-0	$C_{12}H_{12}$	156,22	-15	260	1,0080
Hexadecane (C16)	544-76-3	$C_{16}H_{34}$	226,44	18	287	0,7733
TXIB	6846-50-0	$C_{16}H_{30}O_4$	286,41	-70	280	0,9400

TXIB = 2,2,4-Trimethyl-1,3-pentanediol diisobutyrate
mw: molecular weight, mp: melting point, bp: boiling point

3 Test materials

The following test materials were selected. They had been supplied by the customer.

Sample	**Film**	**Barrier**	**Thickness (µm)**
Sample A	BOPP standard	BOPP	20
Sample C	Nativia NTSS 25	BOPLA	25
Sample D	Extendo XFWL	EVOH	20
Sample K	coated BOPP	PVDC/acrylic	26

A commercially available 12 µm thick film from biaxially oriented PET was added by Fraunhofer IVV as a reference.

4 Method for the permeation test

The films were placed in special permeation cells and stored at the selected temperature. In the lower part of the cells an intentionally contaminated cardboard sample was placed. This cardboard was spiked with the test substances in a way that their concentration was always around 750 µg of substance per g of cardboard. The tested films were in direct contact with the cardboard. The upper part of the cells was separated from the lower part by the samples. This part was

purged with a flow of pure nitrogen. The nitrogen flow transported the permeated substances into the detection unit (see original report for further details). Calibration was performed with injections of known amounts of the substances.

Where possible, permeation rates are reported as their values under stationary conditions, i.e. when they achieved a constant value over time. In the case of highly permeating films, here: sample A, the onset of depletion of the contaminated cardboard already occurred during the measurement period, resulting in a maximum of the measured rate. In this case, the maximum of the observed permeation rate is reported. This means that the permeation rate that would be measured under true stationary conditions might be even higher.

5 Results of the permeation test

All samples were measured in duplicate. Sample A (low barrier, "set 1") was measured at a lower sensitivity for a period of 19 days. The other samples (medium and high barrier, "set 2") were measured at a higher sensitivity for a period of 47 days. The following table shows the permeation rates of the tested films at 40 °C. The last two lines show the detection limits (DL) of the measuring unit.

Permeation rates in (µg/d*dm²)	Dodecane (C12)	Naphthalene	1-Methylnaphthalene	Tetradecane (C14)	1-Ethylnaphthalene	Hexadecane (C16)	TXIB
Set 1							
Sample A	5081	707	1584	1372	992	240	249
	4721	679	1602	1408	1013	237	239
Set 2							
12 µm PET			<0.006		<0.006		0.010
	0.010	0.011	<0.006	0.008	0.007	<0.006	
Sample C			<0.006	<0.006	<0.006	<0.006	<0.009
	0.010	0.008	<0.006	<0.006	<0.006	<0.006	<0.009
Sample D				0.020	<0.006	0.014	<0.009
	0.011	0.009	<0.006		<0.006	0.020	0.0
Sample K	1.51	0.39		4.99			
				5			0.30
DL Set 1	0.218	0.191	0.225	0.243	0.221	0.256	0.368
DL Set 2	0.006	0.005	0.006	0.006	0.006	0.006	0.009

Green highlighted fields: permeation in equilibrium
Yellow highlighted fields: observed maximum of permeation rate (no equilibrium)

6 Discussion of permeation results

Comparison of the high barrier materials EXTENDO XFWL 20 (sample D) and Nativia NTSS 25 (sample C) with PET, 12 µm (reference):

All three materials show permeation rates close to or even below the detection limit. With respect to their very high barrier against mineral oil components, they can be rated as being equivalent.

Comparison of BOPP (sample A) with the high barrier materials EXTENDO XFWL 20 (sample D) and Nativia NTSS 25 (sample C) as well as PET, 12 µm (reference):

Sample A, a 20 µm BOPP film, shows very high permeation rates. They reach a maximum already within some hours for C12 and naphthalene and then decrease as the concentration of the substances in the contaminated card board decreases. In comparison to the high barrier materials sample C, D, and PET as a reference, their permeation rate is higher between a factor of 10000 and 500000, depending on the substance.

Comparison of the high barrier materials EXTENDO XFWL 20 (sample D) and PET, 12 µm (reference) with the PVDC / acrylic coated barrier material (sample K)

Depending on the permeating substance, the permeation rate of sample K is higher by a factor between about 25 and more than 1000 in relation to the high barrier material "sample D". Although sample K already allows for a substantial barrier improvement over the BOPP film, it is significantly inferior to sample D and to the equivalent PET reference.

7 Signature

Fraunhofer Institute
Process Engineering
and Packaging

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Prof. Dr. Horst-Christian Langowski
(Institute Director)

Johann Ewender
(Examiner)