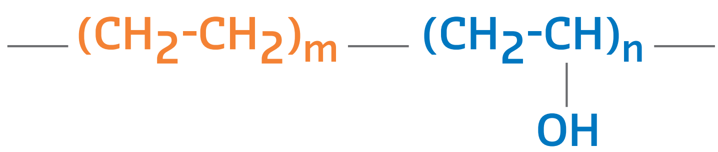
EVAL™ EVOH Resins and Monolayer Film

www.evalevoh.com

EVAL™, a unique Kuraray technology

EVAL™ EVOH combines the excellent gas barrier properties and resistance to organic solvents of polyvinyl-alcohol with the easy processing and water resistance of polyethylene. EVAL™ is commercially available as granules for coextrusion and coinjection, or as monolayer film for lamination. EVAL™ is a crystalline copolymer with the following molecular structure:

Ethylene and Vinyl ALcohol



Characteristics

High gas barrier properties

Extended freshness, less waste

Without an effective gas barrier, oxygen may penetrate packaging and spoil the contents. EVAL™ keeps oxygen out and safeguards quality, extending shelf life and avoiding waste. This cost-saving function is commonly added to food, medical, pharmaceutical, cosmetic, agricultural and industrial packaging applications.

Migration barrier

Improving food safety

EVAL™ provides excellent functional barrier against organic solvents, protecting food against the migration of contamination like MOSH/MOAH mineral oils that can compromise food safety. EVAL™ also resists permeation of hydrocarbons and grease, maintaining packaging appearance. In addition to protecting food, this property also protects the environment, locking chemical substances inside safe and convenient plastic packaging.

Aroma barrier, no flavour scalping

Assuring quality in new forms of distribution

While keeping oxygen and other gases out, EVAL™ also effectively blocks odours, protecting product integrity. EVAL™ locks volatile fragrance and ingredients inside packaging without absorbing them, and preserves aroma until it can be enjoyed by the consumer.

Transparency

A clear and reliable alternative to aluminium foil

All-plastic EVAL™ barrier layers have excellent flex crack and pinhole resistance, even when flexed, folded and shaken during processing and distribution. EVAL™ offers a reliable barrier alternative to Al foil, with the addition of excellent transparency and safe and low-impact energy recovery at end of life.

Energy efficiency

Extending service life

EVAL™ barrier helps prolong performance and service life in vacuum insulation and reliable all-plastic pipe heating/cooling systems.

Fuel vapour barrier

Safety and environmental protection

Light weight barrier plastic tanks and lines improve the performance and safety of automotive fuel systems. EVOH is the only conventional plastic that meets strict international emission standards.

Solvent resistance

Safe protection against permeation of solvents and agricultural chemicals

EVAL™ shows excellent physical resistance to solvents, and barrier against their permeation into the environment. Because of its resistance to absorption and swelling, EVAL™ is typically used as the inner contact layer in UN-approved chemical bottles. A safe way to transport chemical concentrates.

Hydrophilic, anti-static and glossy appearance

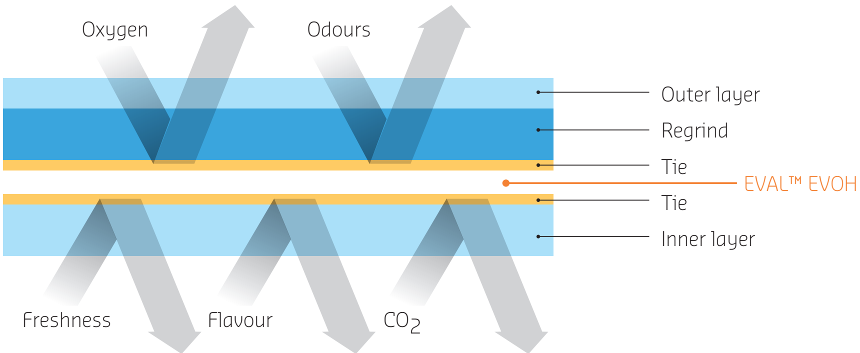
More than just a gas barrier layer

EVAL ™ is a hydrophilic polar material with anti-static properties and a glossy appearance when used as an outside layer. In addition to packaging, its unusual mix of properties leads to use as a technical plastic for industrial, electronic and medical components.

Function, performance and properties

"EVAL™ EVOH resins have outstanding barrier properties, offering performance far greater than any conventional polymer."

Typical coextrusion multilayer structure



Processability of EVAL™ resins

EVAL™ resins are thermoplastic polymers, suitable for use on conventional converting equipment for the following processes:

- monolayer film extrusion (blown or cast)
- multilayer film coextrusion (blown or cast)
- sheet coextrusion
- coextrusion blow moulding
- pipe coextrusion
- extrusion coating
- coextrusion coating
- pipe coextrusion coating
- coinjection moulding
- lamination (EVAL™ monolayer film)

EVAL™ resins can be coextruded with many types of polyolefins, polyamides, polystyrene and polyesters. Downstream processing such as thermoforming, vacuum and pressure moulding and printing can be normally done with flexible or sheet structures containing EVAL™ resins, with special grades available for even the most demanding conditions.

Thin coextruded barrier film structures containing EVAL™ can be laminated to other substrates, adding barrier function to paper, carton and other renewable resources.

Monolayer EVAL™ film can similarly be laminated to virtually any substrate, providing exceptional barrier properties to demanding technical and industrial applications, and as barrier sealant.

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For technical information

Learn more and find your regional contact at www.evalevoh.com
eval.eu@kuraray.com

EVAL™ resin grades

The widest range of grades

A variety of EVAL™ grades have been developed specifically for different methods of production and secondary processing. The perfect balance of required properties is achieved by using Kuraray technology to modify the ratio of ethylene to vinyl alcohol in the copolymer. The result is the widest available range of EVOH grades.

EVAL™ M type
has the lowest ethylene content available, and provides the highest barrier for automotive and flexible applications.

EVAL™ L type
has a very low ethylene content and is suitable as an ultra-high barrier grade in flexible, bottle and sheet applications.

EVAL™ F type
offers superior barrier performance with long-term run stability, and is widely used as the standard grade for flexible, automotive, bottle and tube applications. Specific versions exist for coating and pipe applications.

EVAL™ T type
was specially developed to obtain reliable layer distribution in thermoforming, and has become the industry standard for multilayer sheet and thermoformed flexible applications.

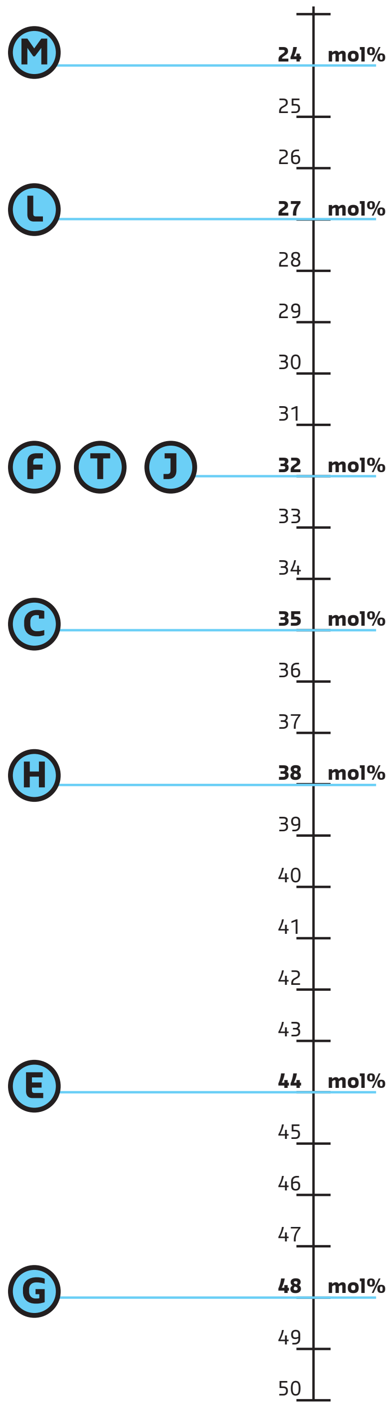
EVAL™ J type
offers thermoforming results even superior to those of T, and can be used for unusually deep-draw or sensitive sheet-based applications.

EVAL™ C type
can be used for high-speed coextrusion coating and cast flexible applications.

EVAL™ H type
combines high-barrier properties and long-term run stability and thermoformability. The higher ethylene content allows easier processing and longer running times on older coextrusion equipment, especially for blown flexible structures.

EVAL™ E type
has a higher ethylene content that allows for greater flexibility and even easier processing.

EVAL™ G type
has the highest ethylene content, making it the best candidate among standard EVAL™ grades for stretch and shrink film applications.



EVAL™ (EVOH) resin grades and typical characteristic values

The following is an overview of EVAL™ resin grades with their typical properties and applications, distinguished by standard grades, grades for specific processing and secondary processing, and special grades. EVAL™ SP grades have improved orientation properties, taking EVOH where it could not go before.

Table 1a: Standard grades

Grade	Et.Cont. (mol%)	Density*1 (g/cm^3)	MFR*2 (g/10min)	Tm (ºC)	Tg*3 (ºC)	OTR*4 ($cm^3.20\mu m/m^2.day.atm$)	Application
L171B	27	1.21	4.0*5	190	63	0.1	ultra high-barrier
F101B	32	1.19	1.6	183	60	0.3	fuel tank, bottle
F171B	32	1.19	1.6	183	60	0.3	bottle, sheet, film, tube
H171B	38	1.17	1.7	172	56	0.7	bottle, sheet, film, tube
E105B	44	1.14	5.5	165	53	1.9	sheet, film, tube
G156B	48	1.12	6.4	157	50	3.7	oriented shrink film

*1 20ºC   *2 190ºC, 2,160g   *3 Dry   *4 20ºCx65%RH, ISO 14663-2 annex C   *5 210ºC, 2,160g

Table 1b: Grades for specific processing conditions

Grade	Et.Cont. (mol%)	Density*1 (g/cm^3)	MFR*2 (g/10min)	Tm (ºC)	Tg*3 (ºC)	OTR*4 ($cm^3.20\mu m/m^2.day.atm$)	Application
F101A	32	1.19	1.6	183	60	0.3	F101 without external lubricant
F104B	32	1.19	4.4	183	60	0.3	high MFR F-type
FP101B	32	1.19	1.6	183	60	0.3	extended pipe service life
FP104B	32	1.18	4.4	183	60	0.3	extended pipe service life
T101B	32	1.17	2.0	183	55	0.4	thermoformed film, sheet
J102B	32	1.17	2.0	183	54	0.6	deep thermoforming, sheet
C109B	35	1.18	8.5	177	53	0.5	extrusion coating
E171B	44	1.14	1.7	165	53	1.9	low MFR E-type

*1 20ºC   *2 190ºC, 2,160g   *3 Dry   *4 20ºCx65%RH, ISO 14663-2 annex C

Table 1c: Special grades

Characteristics	Comparison to standard grades	Grade
Thermoforming grade	Improved EVAL™ layer distribution during deep thermoforming	SP Series, LT Series, J171B
Soft grade	Improved flex-crack resistance with similar barrier	FS201B
High impact strength grade	Improved impact strength	LA170B
Fuel tank grade	Ultra high barrier properties for automotive	M100B
Pipe grade	Provides extended service life at high temperature	FP101B, FP104B, EP105B
Retort grade	Retort flexible grade. Improved retort performance for thin transparent structures.	FR101B, LR171B

Please contact us for more information concerning Special grades.

Table 1d: SP grades. Improved orientation and layer distribution during secondary processing

Grade	Et.Cont. (mol%)	Density*1 (g/cm^3)	MFR*2 (g/10min)	Tm (ºC)	Tg*3 (ºC)	OTR*4 ($cm^3.20\mu m/m.day.atm$)	Application
SP521B	27	1.19	4.1*5	190	63	0.2	improved layer distribution in forming
SP482B	32	1.16	2.0	183	60	0.6	improved film orientation
SP292B	44	1.13	1.9	165	48	3.0	improved film orientation

*1 20ºC   *2 190ºC, 2,160g   *3 Dry   *4 20ºCx65%RH, ISO 14663-2 annex C   *5 210ºC, 2,160g

Processing agents for EVAL™ resin

We also offer the following processing agents, useful for specific EVAL™ resin applications and processing methods

- Recycling agent
- Purging agent

Please contact us for more information.

EVAL™ monolayer film grades

Table 2: EVAL™ EVOH monolayer film grades for lamination

Grade	Type	Thickness (μm)	Ethylene Content (mol%)	Density (g/cm³)	OTR*1 (cm³/m².day.atm) 20°C 65% RH ISO 14663-2	Applications
EF-XL	Biaxially oriented	12	32	1.20	0.4	high barrier
EF-F	Non-oriented	12	32	1.20	0.6	high barrier, deep draw
EF-E	Non-oriented	30	44	1.14	1.0	sealable, deep draw
VM-XL	Biaxially oriented, Aluminium metalized.	15	32	1.20	<0.05	ultra-high barrier
HF-M	Non-oriented	12	-	1.10	-	matt for wallpaper use

Gas barrier properties

1. General information

EVAL™ resins have outstanding gas barrier properties that exceed those of all other plastics used today for barrier purposes.

Fig. 1: EVAL™ layer thickness and Oxygen Transmission Rate

The Oxygen Transmission Rate of an EVAL™ layer is inversely proportional to its thickness. Put another way, by doubling the thickness of the EVAL™ layer, the oxygen transmission is reduced by one half.

Packaging performance can be designed and optimised by selecting the appropriate EVAL™ layer thickness.

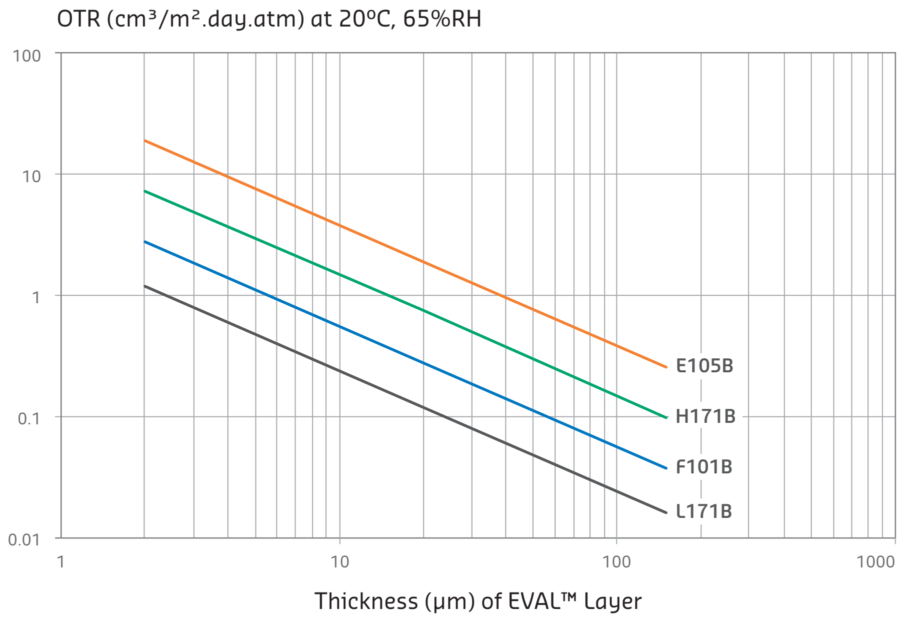


Table 3: Gas Transmission Rates of selected polymers

In addition to oxygen, EVAL™ resins offer outstanding barrier against other gases as well.
Data is provided below for helium and typical MAP gases like carbon dioxide and nitrogen.

Films	Gas Transmission Rates at 0% RH (cm³.20 μm/m².day.atm)							
	H_2 (20ºC)	N_2 (25ºC)	CO_2 (25ºC)	He (25ºC)	Ar (35ºC)	Ar (50ºC)	Kr (35ºC)	Kr (50ºC)
F101B	30*	0.017	0.81	160	-	0.5	-	0.4
E105B	200	0.13	7.1	410	1.6	7.0	-	1.8
OPA	-	12	205	2,000	-	-	-	-
CPA	-	-	-	-	60	150	23	68
OPET	-	8	110	3,100	-	-	-	-
OPP	10,000	730	9,100	-	8,100	28,000	6,900	23,000
LDPE	-	3,100	42,000	28,000	19,000	46,000	25,000	74,000

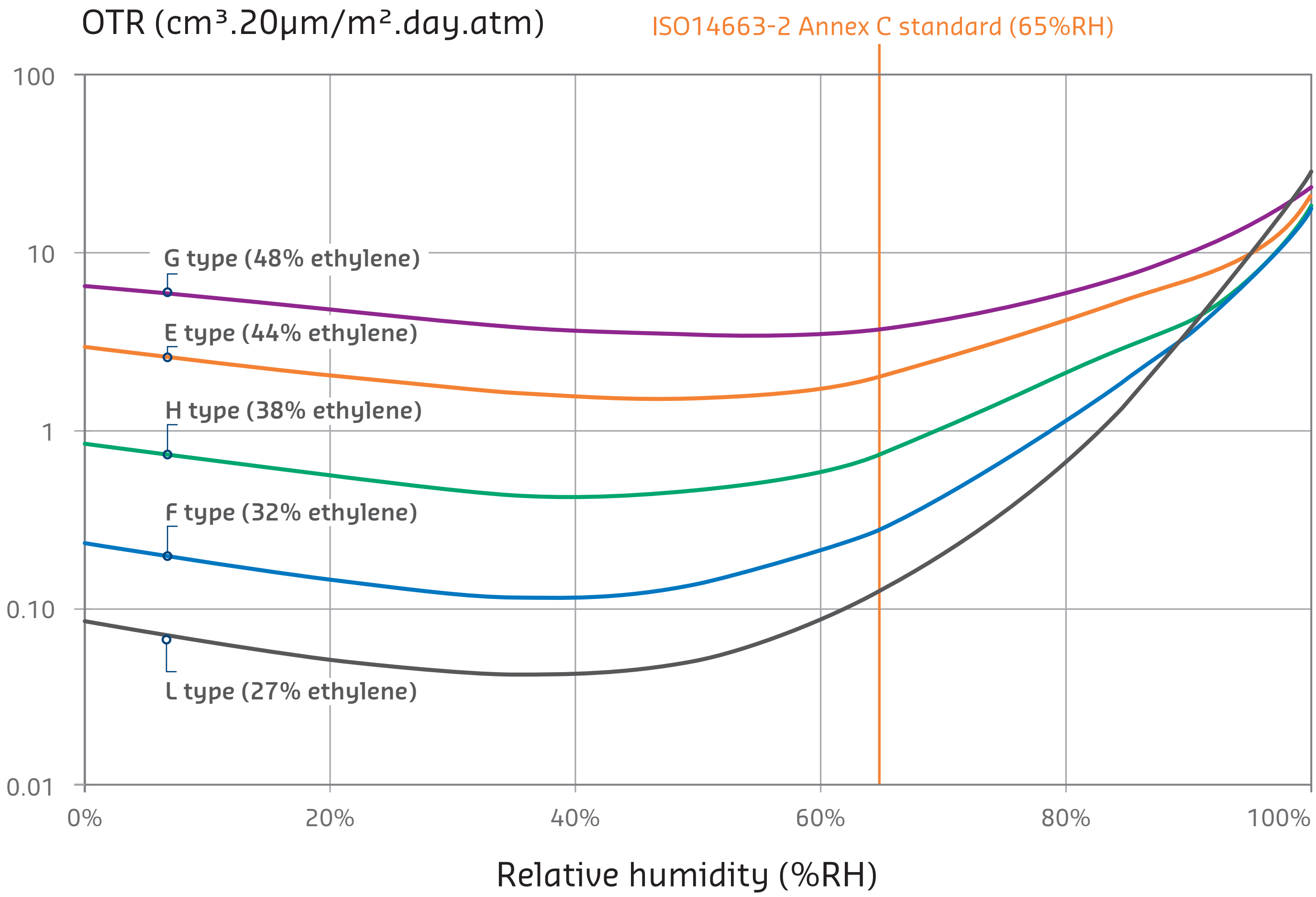
*F171B

"EVAL™ barrier properties work in both directions, keeping unwanted gases and contamination out, while maintaining inside gas mixes longer."

2. Effect of environmental conditions

EVAL™ resins, as indicated by the presence of hydroxyl groups in their molecular structure, are hygroscopic and readily absorb moisture. The amount of moisture that will be absorbed and the speed of absorption will depend upon the environmental conditions encountered. Moisture absorption is dependent upon temperature and the relative humidity of the environment.

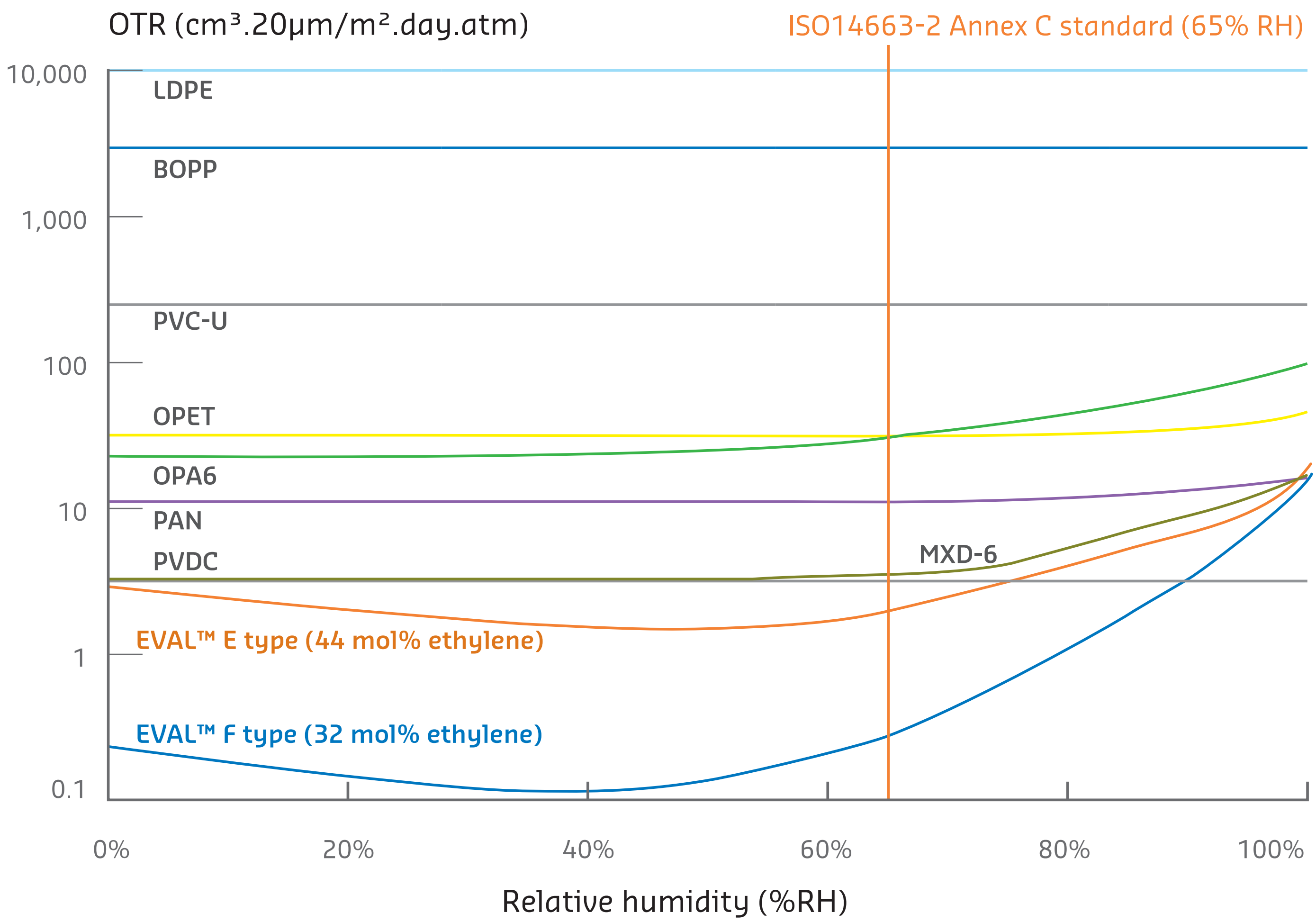
Fig. 2: Oxygen Gas Transmission Rate at 20ºC versus relative humidity in EVAL™ resins



However, though the barrier properties of EVAL™ resins decrease with increasing humidity, even at high humidity, EVAL™ resins still maintain their superior barrier properties when compared with other materials as shown in figure 3.

Furthermore, by coextruding EVAL™ resin between layers of high moisture barrier resins like polyethylene or polypropylene, the loss of oxygen barrier properties is greatly diminished. Nevertheless, humidity should be considered in designing high-barrier structures.

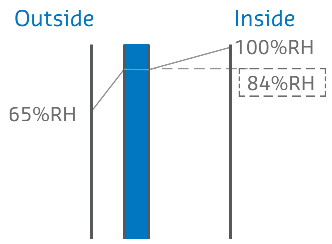
Fig. 3: Oxygen Transmission Rate of various polymers versus relative humidity at 20°C



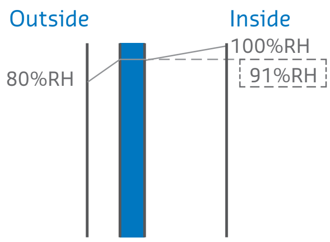
Adjusting structure design for optimal performance

Based on the application and typical conditions of use, it is possible to optimise barrier performance. The equilibrium relative humidity of the EVAL™ layer can be lowered by shifting its location or by carefully choosing the other materials in the structure.

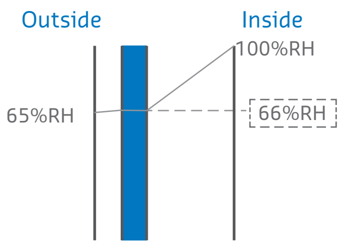
liquid products



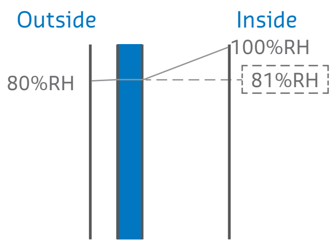
OPP/EVAL™/LDPE
µm 20/15/50



OPP/EVAL™/LDPE
µm 20/15/50

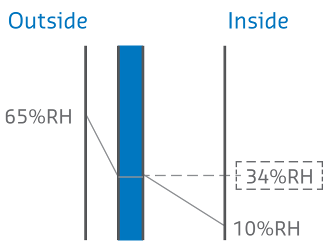


OPA/EVAL™/LDPE
µm 15/15/50

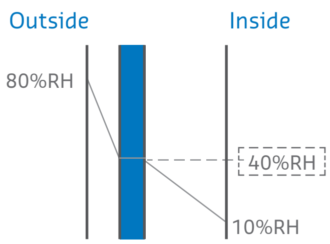


OPA/EVAL™/LDPE
µm 15/15/50

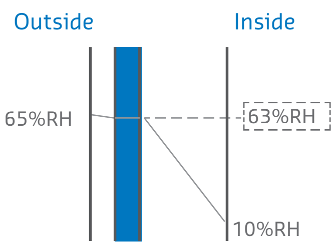
dry products



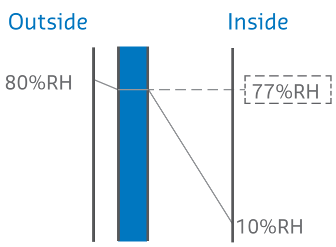
OPP/EVAL™/LDPE
µm 20/15/50



OPP/EVAL™/LDPE
µm 20/15/50



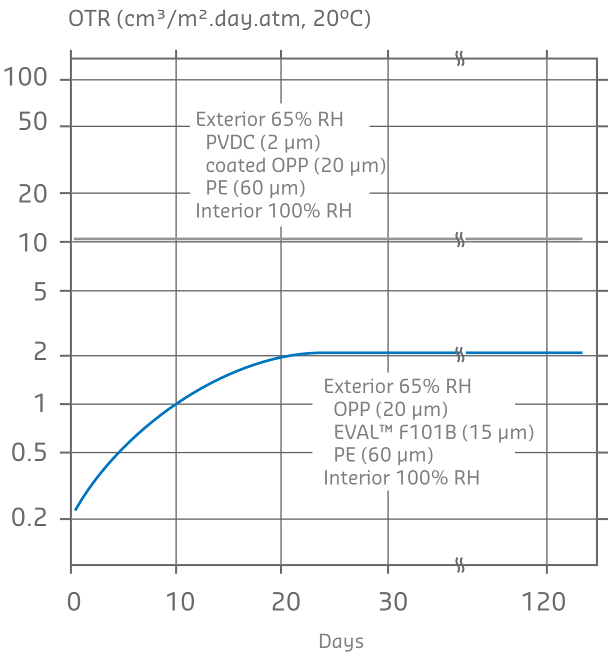
OPA/EVAL™/LDPE
µm 15/15/50



OPA/EVAL™/LDPE
µm 15/15/50

Figure 4 shows that even in packaging wet foods, multilayered structures containing an EVAL™ resin layer can be designed to give 10 times the oxygen barrier properties of PVDC.

Fig. 4: Changes in Oxygen Transmission Rate for composite film structures



With its exceptional barrier properties, at equal thickness EVAL™ EVOH provides better barrier performance than any other conventional polymer. A well-designed team of different layers, each with its own technical strengths, work together to make the most efficient structures possible.

Regrind of multilayer structures is commonly used on stream as a structural layer, and is proven not to disrupt polyolefin recycling streams.

The excellent migration barrier of EVAL™ EVOH ensures content integrity and opens new opportunities for post consumer recycling in food packaging.

Water vapour transmission and moisture absorption of EVAL™ resins

As mentioned in the previous section, EVAL™ resins are hygroscopic and absorb moisture. The amount of moisture that will be absorbed and the speed of absorption will depend upon the environmental conditions encountered. Moisture absorption is dependent upon temperature and the relative humidity of the environment. For comparison, table 5 shows the water vapour transmission rate (WVTR) of monolayer EVAL™ films and films of other polymers. Figure 8 shows the speed of moisture absorption of EVAL™ monolayer film, normally the EVAL™ is co-extruded/laminated with other materials, which will reduce this speed of moisture absorption in the EVAL™ greatly.

Table 4: WVTR of monolayer films

Films	WVTR (ASTM E96-E). 40°C 0/90% RH (g.30μm/m².day)
EVAL™ L type (27% ethylene)	85
EVAL™ F type (32% ethylene)	52
EVAL™ T type (32% ethylene)	37
EVAL™ H type (38% ethylene)	32
EVAL™ E type (44% ethylene)	27
EVAL™ G type (48% ethylene)	23
EVAL™ F101 (bi-axially oriented)	20
Extrudable high barrier PVDC	3
Bi-axially oriented PP	5
HDPE	5
PP	9
LDPE	15
Bi-axially oriented PET	15
Rigid PVC	40
PAN	80
PS	112
Bi-axially oriented PA 6	134

Fig. 5: Moisture absorption of EVAL™

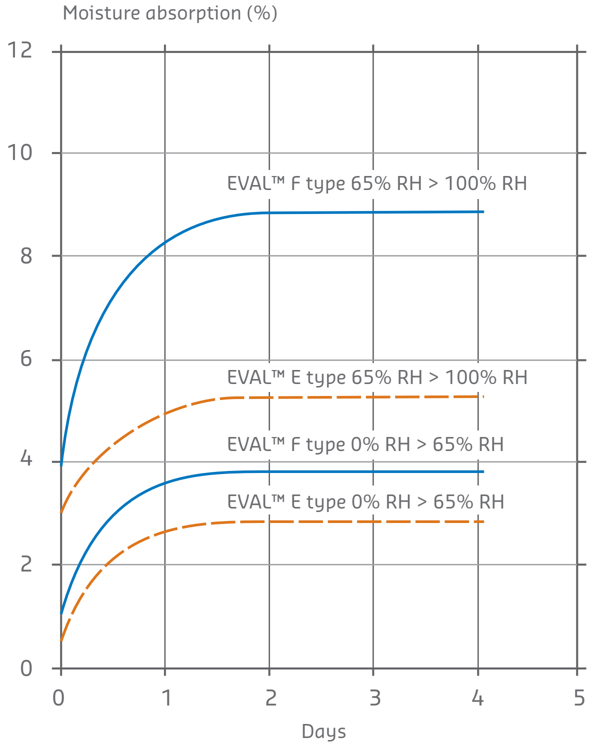


Fig. 6: Equilibrium moisture absorption and relative humidity of EVAL™

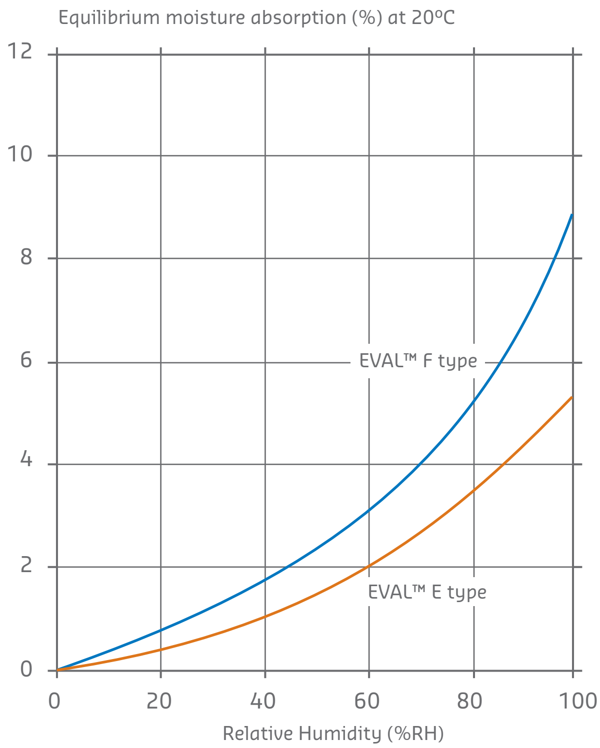
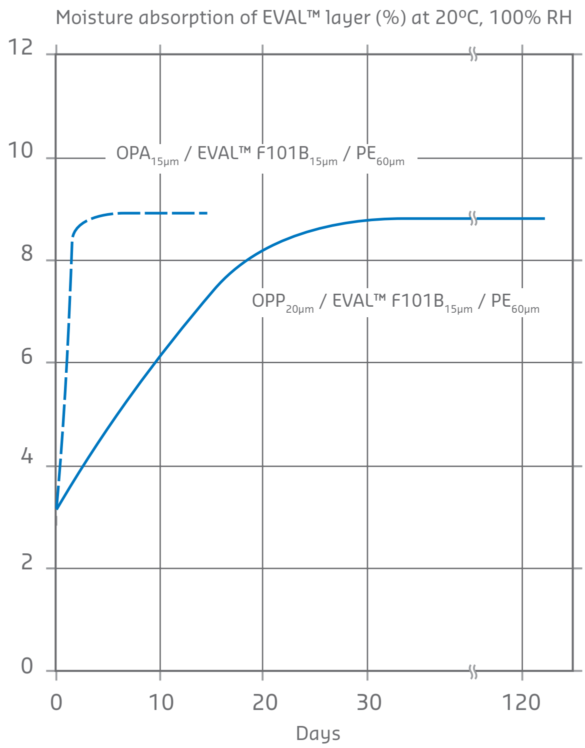


Fig. 7: Moisture absorption of EVAL™ multilayer film as a function of time



Mechanical properties

EVAL™ resins have high mechanical strength, elasticity and surface hardness plus excellent abrasion resistance.

Table 5: Typical mechanical properties of standard EVAL™ resin grades

EVAL™ Grade Name				L171	F171	F101	F104	C109	T101	H171	E105
Ethylene content(%)	mol%			27	32	32	32	35	32	38	44
Tensile stress at break	MPa	ISO 527	Conditioning: 23ºC, dry Measurement: 23ºC, 50%RH	41	34	34	34	32	33	31	29
Tensile strain at break	%	ISO 527		12	14	15	14	14	16	16	18
Young's Modulus	GPa	ISO 527		4.9	4.5	4.5	4.5	4.3	4.3	4.0	3.5
Flexural Modulus	GPa	ISO 178		4.7	4.3	4.3	4.3	4.0	4.1	3.8	3.2
Charpy Impact Strength	kJ/m²	ISO 179-1		6	7	8	7	6	9	8	10
Rockwell Hardness	HRM	ISO 2039-2	Conditioning: 23ºC, dry Measurement: 23ºC, 50%RH	97	92	92	92	89	92	83	80

Thermal characteristics

Table 6: Typical thermal properties of standard EVAL™ resin grades

EVAL™ Grade Name				L171	F171	F101	F104	C109	T101	H171	E105
Melting Temperature	ºC	ISO 11357	Measurement: 10ºC/min	190	183	183	183	177	183	172	165
Crystalization Temperature	ºC	ISO 11357	Measurement: 10ºC/min	164	158	158	158	155	158	151	145
Glass Transition Point	ºC	ISO 11357	Measurement: 20ºC/min	63	60	60	60	58	60	56	53
Vicat Softening Point	ºC	ISO 306	Conditioning: 23ºC, dry Measurement: 23ºC, 50%RH	181	172	172	172	167	165	162	152
MFR	g/10min	ISO1133	190ºC, 2160g	4.0 (210ºC)	1.60	1.60	4.40	8.50	2.00	1.70	5.50
Density	g/cm³	ISO1183-3	20ºC	1.21	1.19	1.19	1.19	1.18	1.17	1.17	1.14

Processing EVAL™ resins

Cylinders

- Natural or shallow grooved is recommended.
- As steel type of cylinders, nitrided steel or special alloys are used for their superior wear resistance with the inner surface honed.
- The outside of the cylinder should be divided into 4 to 5 zones for good extrusion temperature control.
- The lower part of the hopper or the feed throat should be able to be cooled by water to avoid premature melting of the surface of the pellets which might cause bridging and/or blocking of the hopper.

Screws

Extruder properties such as output, resin temperature, consistency of extruded product, output stability, power consumption, etc. are mainly determined by screw design.

- **Screw type**: single stage metering screw (full flight) is recommended.
- **Design L/D**: screws with following length to diameter ratios are preferred:
  - F grade (32 Et%) L/D ratios of at least 26 are desirable
  - E grade (44 Et%) L/D ratios of 24 or larger are recommended
- **Compression ratio**: a compression ratio of 3 is recommended (compression ratio calculated as ratio of channel volume of feed and metering section of screw).
- **Zone distribution**: screws with constant pitch, relative long feed section and a gradually decreasing channel depth in the compression zone leading to a metering zone are recommended; in particular, the following zone distributions will be best suited for processing EVAL™ resins.

Table 7: Recommended zone distributions

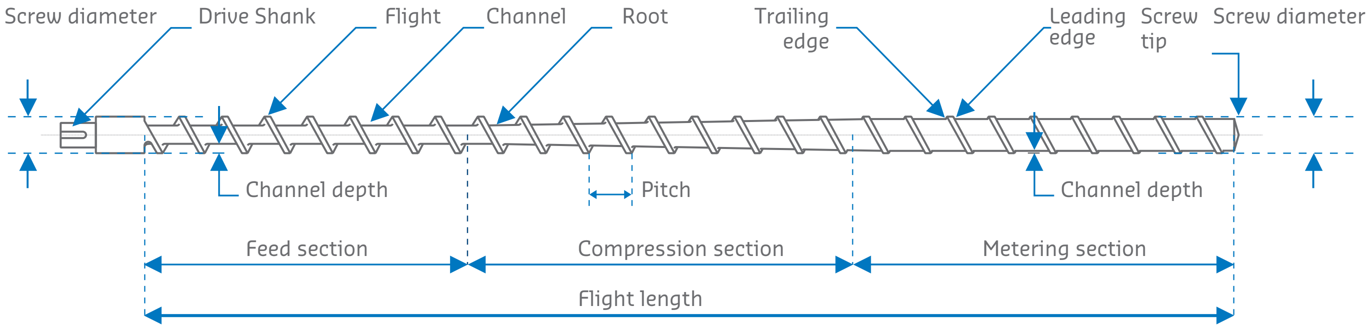
L/D	Feed zoned	Zone distribution Compression zone	Metering zone
28	8D	10D	10D
26	8D	9D	9D
24	8D	8D	8D

Quick compression type screws, (4D or less for compression zone) as used for processing PA, should be avoided.

- **Mixing zone**: screws with mixing zones or heads may be used for extruding EVAL™ though this is generally not recommended. The use of a (high shear) mixing head can restrict the flow of EVAL™ resin, causing degradation of the polymer under prolonged heat and time.
- **Screw tip**: screw tip angle of 120º - 150º is recommended.
- **Flight**: a constant flight width of 0.1D is recommended.

- **Screw material**: chromium molybdenum steel, normalised prior to plating is recommended as screw material. To help prevent the build-up of polymer on the screw, surface hard chrome plating (30 - 50 µm) with buff finishing is recommended, though in some cases nitration treatment can be applied as well.
- **Screw clearance**: instead of recommendation, following example is given for screw clearance: inside diameter of cylinder is 60.02 - 60.05 mm in combination with screw diameter of 59.87 - 59.89 mm. When, due to wear, the screw clearance becomes too large, melt backflow can occur which can cause material degradation due to high shear rates.

Fig. 8: Typical screw design for 60 mm single stage metering screw



Note: screw drawing is not scaled to dimensions

Typical dimensions

Diameter	60 mm		
Flight length	1 560 mm (26D)		
Feed section length	480 mm (8D)		
Compression section length	540 mm (9D)		
Metering section length	540 mm (9D)		
Compression ratio	3		
Constant pitch (flight lead)	60 mm (1D)		
Channel depth feed section	8.4 mm		
Channel depth metering section	2.5 mm		
Channel width	54 mm (0.9D)		
Flight width	6 mm (0.1D)		
Flight angle	17.65º		
Screw tip angle	120º ~ 150º		
Flight to root radius	Feed section	Compression section	Metering section
• Leading edge radius	8.4 mm	8.4 - 2.5 mm	2.5 mm
• Trailing edge radius	5 mm	5-2 mm	2 mm

Typical output

- For single flight metering screw, typical output can be calculated with following simplified method, where the drag flow, pressure flow, etc. are not considered.

$$Q = \tfrac{1}{2} \bullet \rho \bullet N \bullet \pi \bullet D \bullet H_m \bullet W \bullet \cos^2 \phi \bullet 60 \bullet 10^{-6}$$

where : Q = output (kg/hr)
ρ = melt density (g/cm^3)
N = screw rotation (rpm)
D = screw diameter (mm)
H_m = channel depth in metering zone (mm)
W = channel width (mm)
ϕ = flight angle (deg)

- When the flight pitch (P) equals the screw diameter $(P = \pi.D.\tan\phi \rightarrow \tan\phi = \frac{1}{\pi} \rightarrow \phi = 17.65^{o})$ and W = 0.9D, above equation becomes:

$$Q = 7.7 \bullet 10^{-5} \rho \bullet N \bullet D^2 \bullet H_m$$

which coincides with the experimental results given following conditions:
- back pressure from 0 - 20 MPa
- use of lubricated EVAL™
- ratio actual output / theoretical output ≅ 0.8 - 1 : normal extrusion
- or ratio actual output / theoretical output > 1 : overpacking

- Generated shear rates can be calculated with $\gamma = \frac{1}{60} \bullet \frac{\pi \bullet N \bullet D}{H_m}$ (1/s) and should be in the range of 50 - 100 (1/s)

Table 9 shows typical outputs, achieved using a metering screw with configuration as recommended for EVAL™ resins.

Table 8: Calculated output for metering screw

Screw diameter (mm)	25	40	50	60	90
L/D	26	26	26	26	26
Constant pitch (mm)	25	40	50	60	90
Feeding zone, depth	8D, 4.9 mm	8D, 6.1 mm	8D, 6.6 mm	8D, 8.4 mm	8D, 11.6 mm
Compression zone	9D	9D	9D	9D	9D
Metering zone, depth	9D, 1.4 mm	9D, 1.8 mm	9D, 2.0 mm	9D, 2.5 mm	9D, 3.5 mm
Compression ratio*	3	3	3	3	3
Screen pack (mesh)	50/100/50/50	50/100/50/50	50/100/50/50	50/100/50/50	50/100/50/50
Motor capacity (kW)	2.2 ~ 3.7	7.5 ~ 11	11 ~ 15	15 ~ 22	37 ~ 55
Screw rotation (rpm)**	30 - 70	30 - 70	30 - 70	30 - 70	30 - 70
Output (kg/hr)**	2.1 - 5.0	7 - 16	12 - 29	22 - 51	69 - 162
Shear rate (1/s)***	28 - 65	35 - 81	39 - 92	38 - 88	40 - 94

* volumetric compression ratio ** normal range *** theoretical values

Screen pack - breaker plate

A screen pack and breaker plate, placed between the extruder and die, is common practice in commercial polymer extrusion. The use of a screen pack is recommended for extrusion of EVAL™ resins. The screen pack mesh should be based on commercial polymer extrusion knowledge and common sense. Examples of typical combinations of stainless steel meshes are: 50/100/50/50, 50/100/150/100 or 80/150/50/50.

Typical distance between screw tip and screen pack is 5 to 10 mm, longer distances may cause unnecessary longer residence time. For the breaker plate it is recommended that the holes have a diameter of approx. 5 mm and that the outermost array of holes inscribes the inner surface of the cylinder.

Melt path (adapter, melt pipe)

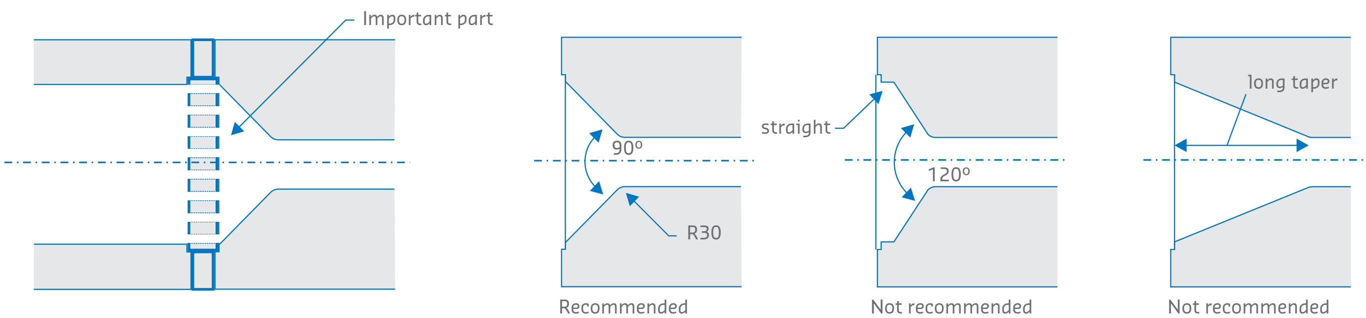
EVAL™ resins are highly adhesive to metal surfaces. If the EVOH extrusion system consists of concave or convex parts, acute angles, etc. EVAL™ resin can easily become restricted in these dead spot areas. Even in a passage of fixed diameter, retention can occur on the wall if the diameter is too large for the flow and therefore the shear rate is too low. Retained resin, exposed to heat at prolonged time, can deteriorate forming gels or oxidised particles. The degraded resin will manifest itself as yellowish, brown or black gels.

When designing process equipment for EVAL™ resins, the following points are recommended for the EVAL™ melt path:

- Shear rate at wall: higher than 6 s^{-1}.
- Average flow rate: higher than 1 cm/s.
- Eliminate any concave, convex, or acute angles in the melt path.
- Minimise the diameter of all adapters.
- Chrome plate (and buff finish) the surfaces exposed to the EVAL™ melt path.

Following are examples recommendable, respectively not recommendable, for the adapter direct after the breaker plate.

Fig. 9: Adapter design



Dies

EVAL™ resins are suitable for normal feedblock type die designs. Special die designs are not necessary, though care should be taken to streamline the flow channels as much as possible, especially with complicated die designs (e.g. for blown film lines) which could have increased number of dead spots and prolonged residence time. As with the adapter and melt pipe, it is recommended to chrome plate the surfaces exposed to the EVAL™ melt.

Lubricant mixed EVAL™ resin

Use of lubricant mixed EVAL™ resin is recommended for improving the stability of the resin flow at the feeding zone of the screw, resulting in a more constant output rate and lower energy consumption of the extruder motor.

Extrusion temperature

In processing any polymer it is important that a homogeneous, completely melted and well mixed mass at a uniform temperature is obtained. Also, the temperature must be well controlled to minimise thermal decomposition of the polymer being processed. EVAL™ resins are no exception to this rule. Upper and lower extrusion temperature limits are as follows:

Table 9: Upper and lower extrusion temperature limits

EVAL™ resin grade		L171	F171	F104	T101	H171	E105
Upper temperature	ºC	240	240	240	240	240	250
Lower temperature	ºC	210	200	200	200	200	185
Melting point	ºC	191	183	183	183	175	165

It should be remembered that when extruder temperature exceeds the recommended upper limits, the polymer may decompose resulting in gels, voids, in the extrudate. On the other hand, at low extrusion temperatures, the resin may only be partially melted, the melt not mixed or heterogeneous, resulting in poor extrudate appearance, erratic web gauge control or unmelted specs in the extrudate.

Coextrusion with polymers such as PET, PA, PC or PP may cause the EVAL™ resin to come in contact with melt streams that are hotter than the recommended maximum temperature. This contact is usually for a short period of time and adverse effects are not encountered.

Table 11 shows typical extrusion temperature conditions for the different EVAL™ grades.

Table 10: Typical screw design characteristics

Extruder diameter	60 mm
L/D	26
Flight	Full flight
Screw pitch	60 mm, constant
Feed zone, channel depth	8D, 8.4 mm
Compression zone	9D
Metering zone, channel depth	9D, 2.5 mm
Compression ratio	3.0
Motor capacity	22 kW
Screen structure	50/100/50/50 mesh

Table 11: Typical Temperature settings for extruding EVAL™ resins

EVAL™ resin grade			M100	L171	F101 FP101	F171	F104 FP104	T101 J102	C109	H171	E105 EP105	E171	G156
Barrel temperature	C_1	ºC	190	190	180	180	180	180	180	175	170	170	165
	C_2	ºC	210	205	200	200	200	200	200	195	190	190	185
	C_3	ºC	215	210	205	205	205	205	205	205	195	195	190
	C_4	ºC	220	215	215	215	215	215	215	215	205	205	200
	C_5	ºC	220	220	220	220	220	220	220	220	210	210	205
Adapter temperature	AD_1	ºC	220	215	215	215	215	215	215	210	195	195	190
	AD_2	ºC	220	215	215	215	215	215	215	210	195	195	190
Die temperature		ºC	215	215	215	215	215	215	215	210	195	195	190

Start-up, purging and shutdown procedures

Start-up

When starting up the extrusion of EVAL™ resins, the following procedure is recommended:

1. The extruder is filled with low MFR (0.7 - 1.0)(*) LDPE in order to avoid oxidisation of residual material in the extruder.
2. Start the processing on a well-cleaned machine by raising the temperature to the set point and introducing LDPE with an MFR of 0.7 – 1.0. Be careful not to start the screw rotation before all LDPE material is heated up to processing conditions.
3. When the extrusion gets stable, directly switch over to EVAL™ without running the extruder empty to avoid oxidisation of EVAL™ by the oxygen in the heated barrel.

Purging

When purging between production campaigns, the following procedure is recommended:

1. Remove EVAL™ from the hopper of the extruder.
2. Introduce LDPE with low MFR (0.7 - 1.0) and purge the EVAL™ out of the extruder while keeping the same process temperature settings (or temperatures slightly lowered). Adjust the processing conditions in case the extrusion becomes unstable. Pressure in the extruder should be high enough to ensure proper cleaning.
3. Continue purging by low MFR LDPE until the residual EVAL™ is completely cleaned out. If this cannot be determined by visually checking product appearance, set up a specific purge test to determine the time or amount of purge material needed to clean the extruder and die head.

Shutdown

1. Introduce LDPE with low MFR (0.7 - 1.0) and purge out EVAL™ from the extruder while keeping the same process temperature settings (or temperatures slightly lowered). Adjust the processing conditions in case the extrusion becomes unstable. Pressure in the extruder should be high enough to ensure proper cleaning.
2. Continue purging by low MFR LDPE until EVAL™ is completely cleaned out by checking product appearance.
3. Screw revolution can be stopped when the extruder is completely filled with low MFR LDPE followed by lowering the extruder temperatures. (In that way, the oxidisation of residual EVOH in the extruder will be avoided).

It is strongly recommended **not** to use PA as a purge material, since PA will react/bond with the residual EVAL™ and form numerous gels.

It is also recommended **not** to use PP, HDPE or adhesive resins as a purge material because some PP and HDPE grades may contain some residual catalyst that can very strongly deteriorate EVAL™.

(*) MFR: Melt Flow Rate mentioned in this section are data measured with a standard melt indexer at 190ºC, 2.16 kg (EVAL™ F101B: 1.6 g/10 min; EVAL™ H171B: 1.6 g/10 min; EVAL™ E105B: 5.5 g/10 min).

Kuraray's ETC-103 purging material

ETC-103 (MFR: 1.0) is a LDPE-based purge resin, developed by Kuraray for purging extruders running EVAL™ resins and for improved transitioning from EVAL™ extrusion to PA or polyolefins. Because of its chemical properties (besides mechanical cleaning), it is used to remove EVAL™ residues from the extruder and die head or to improve current purging procedure.

If time to start-up is critical, a LDPE with higher MFR (5 – 7) can be introduced after purging with the low MFR (0.7 – 1.0) LDPE. The higher MFR (5 – 7) LDPE remains in the extruder after shutdown and can be removed much faster by EVAL™ resin after start-up.

In case the application involves a regrind layer (incl. EVAL™), it is recommended also to purge this extruder with the original material (LDPE, HDPE, PP).

Temporary extruder shutdown

When the extrusion operation has to be stopped temporarily, the following procedures are given as an indication:

Shutdown time	Procedure
Up to 30 minutes	Maintain temperature settings, screw rotation can be stopped
Up to 3 hours	Maintain or lower temperature settings by about 20ºC and operate screw slowly
For more than 3 hours	Purge out EVAL™ as recommended in extruder purging procedure

Please note that maximum shutdown times with EVAL™ in the extruder depend on the design of the processing equipment, temperature settings and residence times.

Polymer change over

The table below lists the recommended purge sequences for change over involving EVAL™ resins.

Resin before change	Resin after change	Sequence
LLDPE, LDPE	EVAL™	Direct
EVAL™	LLDPE, LDPE	Direct
PA, HDPE, PP, PS	EVAL™	PA, HDPE, PP, PS → LDPE → EVAL™
EVAL™	PA, HDPE, PP, PS	EVAL™ → LDPE → PA, HDPE, PP, PS

Prevention of moisture absorption, drying

As mentioned in section 4, EVAL™ resins are hygroscopic and absorb moisture when exposed to atmosphere. Depending upon the fabrication process being used, an increase in the moisture content of EVAL™ resins can cause difficulties in processing; foaming, voids and gels can occur at increased moisture levels (normally above 0.4 wt%).

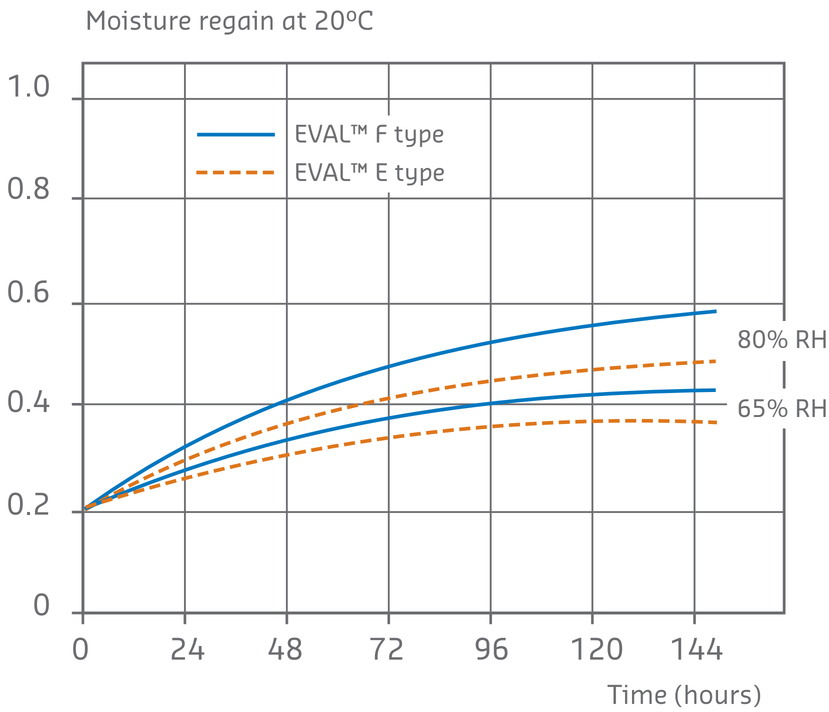
After the production, EVAL™ resins are dried and packaged in moisture proof 25 kg bags or 700 kg octabins. As packaged, the moisture content is controlled to less than 0.3%. This means that EVAL™ resins do not need to be dried, when processed directly after opening the package.

Precautions should be taken after opening a package to prevent against excessive moisture absorption especially in hot humid environments. These precautions include:

- Re-close the package tightly after use.
- If an air conveying system is used, prevent against excessive moisture in the conveying air by use of a moisture trap.
- When using octabins, it is not necessary to open the entire liner when inserting the conveyer pipe; simply cut a hole in the liner and insert the conveyer pipe.

Under normal humidity conditions, packages may be left opened while being used for several days, for more humid conditions see figure 10.

Fig. 10: Moisture regain vs. time



In the event the EVAL™ package has been left open for a longer period and/or at higher humidity conditions, re-drying process such as 3 to 4 hours in hopper dryer or circulating hot air dryer at 90 - 100ºC is recommended. Please make sure that dryer temperature does not exceed 110ºC to avoid discoloration of the EVAL™ resin.

Adhesive resins

To improve the properties of plastic packaging materials, two or more polymer layers in a composite structure are typically employed. These multilayer structures can be prepared by coating, laminating, or coextrusion. When different polymer layers are used in multilayer structures, poor adhesion between the layers typically exists. In order to overcome this deficiency, speciality adhesive resins have been developed. These tie resins act as the glue substance between the non-adhering polymer layers.

With respect to EVAL™ resins, good adhesion can be achieved between EVAL™ resin and PA without using an adhesive layer. In coextrusions with polyolefins, PET, PS, PC, etc., however, an adhesive layer is necessary between these polymers and EVAL™ resins. Depending on the counter resin, different adhesive resins are available on the market.

Utilisation of regrind

One of the serious economic concerns in coextrusion is the loss of multilayer film trim or flash. In monolayer film operations such trim is usually reground and recycled with little economic loss to the processor. With thermally sensitive or vastly different polymers, however, multilayer films or structures cannot be reprocessed.

Such is not the case when using EVAL™ resins. Multilayer structures containing EVAL™ resin can be recovered and reused. For example, coextrusion trim containing EVAL™ resin can effectively be recycled when producing sheets, bottles and fuel tanks.

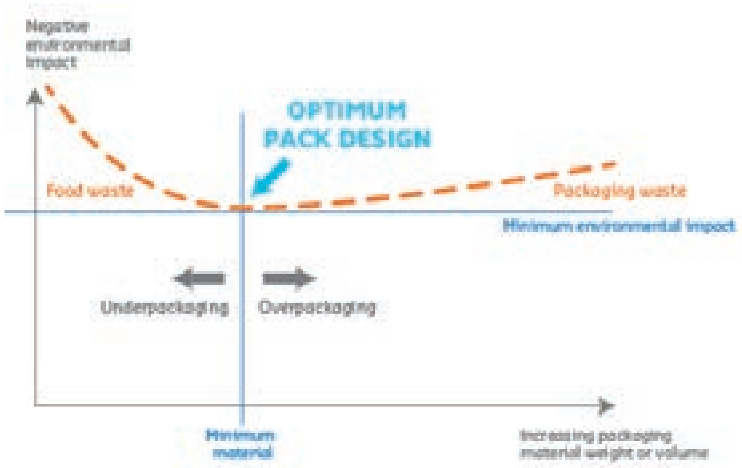
It should be remembered that if the regrind is stored unused for a prolonged period of time, moisture absorption may occur in the EVAL™ component. The regrind then must be dried prior to extrusion. If possible, it is recommended that regrind containing EVAL™ should be processed and reused as soon as possible to avoid potential processing difficulties due to high moisture content.

For several applications, special master batches for regrind are developed by Kuraray to prevent processing troubles during extrusion of this regrind, increase the maximum level of EVOH content in the regrind and enhance the properties of the final product.

Environmental benefits
of EVAL™ resins and monolayer film

Wasting food and commercial products generates its own environmental impact, but also wastes all the resources used to produce, process, package and transport the goods as well. The trick to optimising packaging design is to find the perfect balance between protective function and minimum material use. To be truly effective, a wide view of the entire product life cycle is necessary. Ideally, the materials themselves should have low environmental footprints, but not solve problems at one life cycle stage only to create different problems at another.

A one millimetre thickness of EVAL™ EVOH has about the same gas barrier properties as ten metres of LDPE. With such high performance, EVAL™ layers of only a few microns can add real function to multilayer structures. Barrier performance previously only available from metal or glass can thus be added to lightweight structures based on other recyclable and energy recoverable plastics, or renewable resources like PLA and paperboard.



Protecting quality and value

Although product development tends to focus specifically on EVAL™'s functional barrier properties, EVAL™ helps conserve resources and avoid waste throughout a product's life cycle. Reducing waste, and thus avoiding the loss of all resources invested in the production and distribution of fresh food, is the best way to reduce environmental impact.

When used in laminated structures for food packaging, very thin EVAL™ layers provide the valuable barrier function to the entire structure, usually allowing a decrease in the total amount of packaging materials used. Optimized portion size, lightweight and extended freshness help improve the efficiency of storage, transport and display, saving costs and preserving resources.

Efficient energy use

EVAL™ helps maintain the efficiency of insulation and heating systems, saving money and resources by extending their service life. EVAL™ barrier is effective against new fuels, and blocks emissions from light weight plastic tank and line systems.

Recyclable and recoverable

EVAL™ EVOH is recyclable as post-consumer plastic waste, and will not disrupt polyolefin or PET recycling streams.

EVAL™ has excellent and safe energy recovery properties, often reducing the amount of extra fuel necessary for energy generation from the thermal disposal of sorted waste. Under perfect combustion, the few microns of EVAL™ in the package emit only small amounts of CO_2 and water vapour.

Open innovation

Application development with the world's barrier experts

Kuraray is a world leader in specialty chemicals and functional materials. We are committed to developing products that ensure quality and value while helping our business partners differentiate themselves from their competition.

Kuraray Co. Ltd. was the first company in the world to produce and commercialise EVOH, starting in Okayama, Japan in 1972. Today Kuraray is the world's largest producer of Vinyl Acetate Monomer derivatives, and is still the leader in EVOH technology, production, structure analysis and technical development.

EVAL™ production began in Houston, USA in 1986, and in Antwerp, Belgium in 1999.

Technical centres were established in Kurashiki, Japan, at the Houston and Antwerp production sites, and in Singapore. At each site we work together with converters and end users, bringing our expertise in barrier technology and structure optimisation.

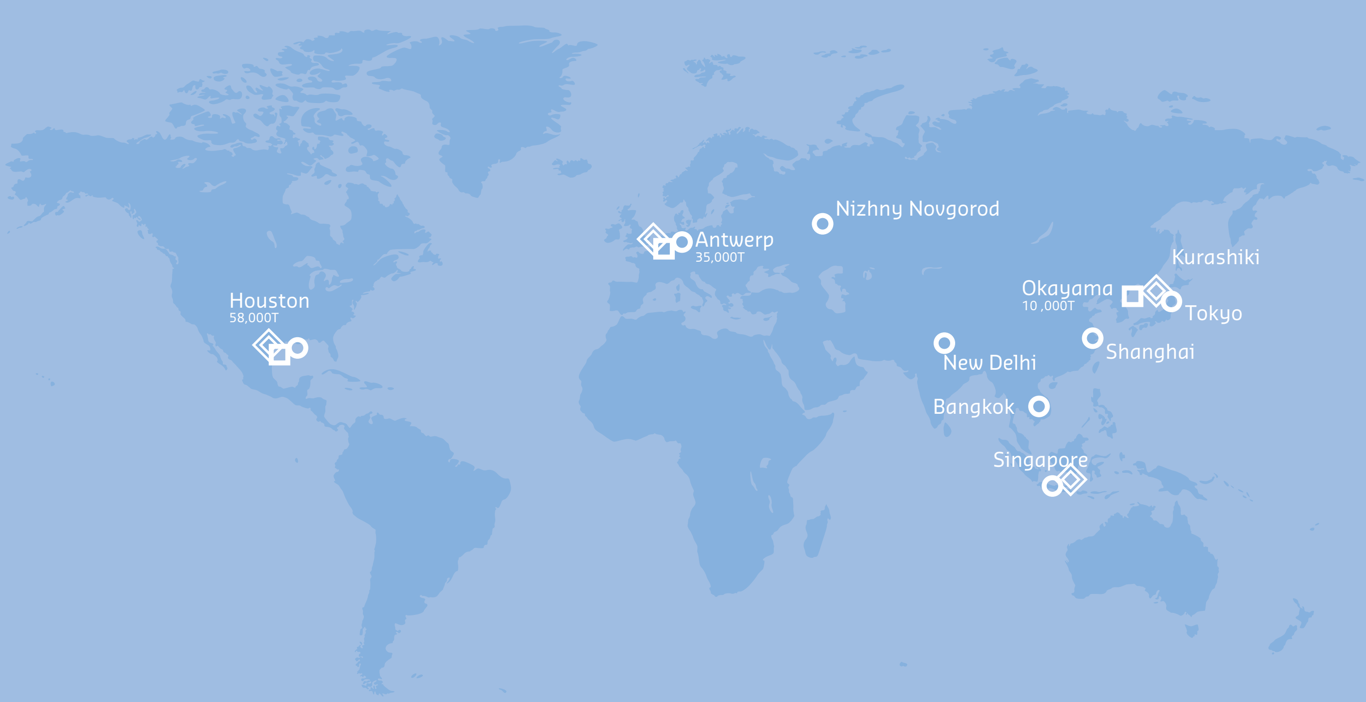
To learn more about our open innovation development, visit our global website **www.evalevoh.com**

HOUSTON, USA

ANTWERP, BELGIUM

SINGAPORE

KURASHIKI, JAPAN



EVAL™ the world's leading EVOH

Asia-Pacific
Kuraray Co., Ltd. (Okayama, Japan)
Capacity: 10,000 tons/year
The world's first EVOH production facility

Americas
EVAL Company of America (Pasadena, Texas, USA)
Capacity: 58,000 tons/year
The world's largest EVOH production facility

Europe
EVAL Europe nv (Antwerp, Belgium)
Capacity: 35,000 tons/year
Europe's first and largest EVOH production facility

www.evalevoh.com

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EVAL™ resins are produced worldwide under unified Kuraray product and quality specifications.