Two-pass sponge weatherstrip processing guide

NORDEL™ 6555 OE EPDM

Product description

NORDEL™ 6555 OE EPDM is an amorphous grade of ethylene propylene diene terpolymer (EDPM) designed for extruded foam applications. It is ideally suited for extruded sponge profiles requiring fast curing conditions. The combination of high viscosity and low crystallinity allows for good balance of processability, extensibility, low temperature properties, and performance. The NORDEL™ 6555 OE EPDM product features improved mixing and extrusion characteristics, suitable for class A surface manufacturing, with improved shape stability of the green compound.

Main characteristics:

- Amorphous
- High diene
- High molecular weight
- Good low temperature properties
- Complies with U.S. FDA 21 CFR 175.105 (consult regulations for complete details)

Applications:

- Extruded profiles (dense or sponge)
- Coolant hoses

Table 1: ASTM & ISO properties[1]

Property	Nominal value	Units	Test method
Specific gravity	0.862	g/cc	ASTM D297
Mooney viscosity (ML 1+4 at 125°C)	55	MU	ASTM D1646
Ethylene content	53.0	wt%	ASTM D3900
Ethylidene norbornene (ENB) content	8.5	wt%	ASTM D6047
Ash content	< 0.1	wt%	ASTM D296
Molecular weight distribution	Medium		Dow method
Propylene content	38.5	wt%	ASTM D3900
Oil content	20-23	phr	Dow method
Residual transition metal	< 10	ppm	Dow method
Volatile matter	< 0.40	wt%	Dow method

[1] Data per tests conducted by Dow. Additional information available upon request. Properties shown are typical, not to be construed as specifications. Users should confirm results by their own tests.

Model formulation

NORDEL™ 6555 OE EPDM Rubber was utilized to successfully produce closed cell sponge weatherstrip profiles. Three different sponge profile formulations were developed to enable flexibility in equipment and processing as well as to meet customer regulatory needs. Those three sponge formulations feature the master batch shown in Table 2 and use curing/blowing agent packages based on: (1) 100% Azodicarbonamide (ADC), (2) a blend of ADC and 4-4'-Oxydibenzenesulfonyl hydrazide (OBSH), and (3) low nitrosamine. The final batch formulations appear in Table 3 (page 2).

Table 2: Master batch formulation

Material	Formulation (phr)
NORDEL™ 6555 OE	120.0
Sunpar 2280	70.0
Calcium carbonate	50.0
N-550 Carbon black	90.0
PEG 4000	2.0
Stearic acid	1.5
Zinc oxide	5.0
Total parts	338.5

Table 3: Final batch formulations

Material	100% ADC formulation	ADC + OBSH blend formulation	Low nitrosamine formulation
	phr	phr	phr
Master batch	338.50	338.50	338.50
Azodicarbonamide (ADC)	4.50	3.50	4.50
4,4-Oxydibenzenesulfonyl hydrazide (OBSH)	–	1.50	–
Calcium oxide (80% dispersion)	2.00	2.00	2.00
Sulphur (80% dispersion)	1.88	1.88	1.88
2-2'-Dithiobis(benzothiazole) (MBTS) 70%	1.00	1.00	1.00
Mercaptobenzothiazole (MBT) 80%	1.50	1.50	1.50
Zinc dibutyldithiocarbamate (ZDBC) 80%	3.60	3.60	–
Tetramethylthiuram disulfide (TMTD) 75%	1.00	1.00	–
Tellurium diethyldithiocarbamate (TDEC) 80%	0.15	0.19	0.19
TetraBenzyl thiuram disulfide (TBzTD) 70%	–	–	1.20
Zinc DiBenzyldithiocarbamate (ZnBEC) 70%	–	–	2.00
Zinc dibutyldithiophosphate (ZnBPD) 50%	–	–	2.50
Total parts	354.13	354.67	355.27

Mixing equipment

A rubber internal mixer with a net volume of 135 L equipped with an intermeshing rotor design and hydraulic ram control was used in this evaluation. However, other rubber mixing equipment can be utilized with appropriate mixing procedures and mixing conditions. See list below:

- Rubber internal mixer: intermeshing rotor design
- Rubber internal mixer: tangential rotor design
- Rubber kneader
- Two-roll mill

Master batch mixing procedures

The following mixer settings and mixing procedure were utilized for master batch mixing:

- Fill factor = 0.73
- Rotor temperature = 120°F (49°C)
- Mixer body temperature = 120°F (49°C)
- Side temperature = 120°F (49°C)

For detailed steps, see Table 4.

Table 4: Master batch mixing procedures

Step	Command	Logic	Time (s)	Temp. (°F [°C])	Rotor RPM
1	Raise ram	None	–	–	30
2	Charge black/ White/Oil	None	–	–	30
3	Charge EPDM	None	20	–	30
4	Close hopper door	None	–	–	30
5	Lower ram	TIME or TEMP	200	194 [90]	30
6	Raise ram	TIME	10	–	30
7	Lower ram	TEMP	–	257 [125]	30
8	Discharge warning	TIME	3	–	30
9	Open drop door	TIME	45	–	30
10	Close drop door	None	–	–	30

Table 5: Final batch mixing procedures

Step	Command	Logic	Time (s)	Temp. (°F [°C])	Rotor RPM
1	Raise ram	None	–	–	15
2	Auto charge (half master batch)	None	20	–	15
3	Close hopper door	None	–	–	15
4	Lower ram	TIME	20	–	15
5	Auto charge (half master batch and curatives)	None	20	–	15
6	Close hopper door	None	–	–	15
7	Lower ram	TEMP	–	140 [60]	15
8	Raise ram	TIME	10	–	15
9	Lower ram	TEMP	–	185 [82]	15
10	Discharge warning	TIME	3	–	15
11	Open drop door	TIME	60	–	20
12	Close drop door	None	–	–	20

Final batch mixing procedures

The following mixer settings and mixing procedures were utilized for final batch mixing:

- Fill factor = 0.72
- Rotor temperature = 73°F (23°C)
- Mixer body temperature = 73°F (23°C)
- Side temperature = 73°F (23°C)

For detailed steps, see Table 5 (page 2).

Master batch mixing curves

Mixing curves for the master batch are included in Figure 1. Fast mixing cycles (<3 min) can be achieved with this model formulation and mixing procedure. Good mixing power (400 kW) was also achieved, enabling good filler dispersion.

Final batch mixing curves

Good temperature control and fast mixing were achieved using the model formulations and mixing procedures described above for all final batch formulations (Figures 2-4).

Figure 1: Master batch mixing curves

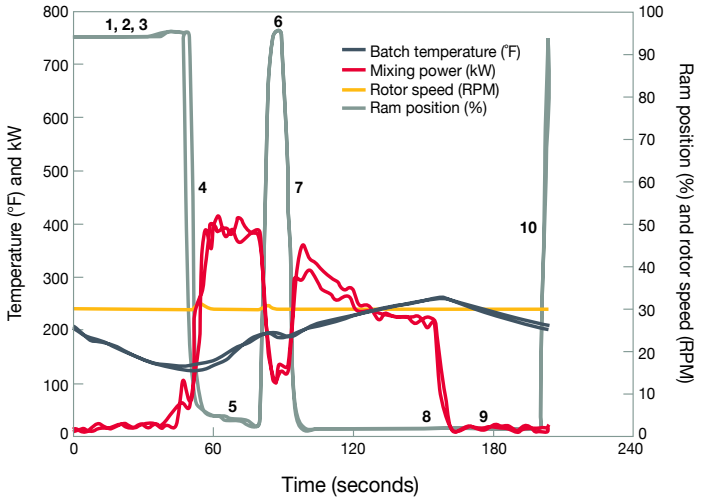


Figure 2: Final batch mixing curves for 100% ADC formulation

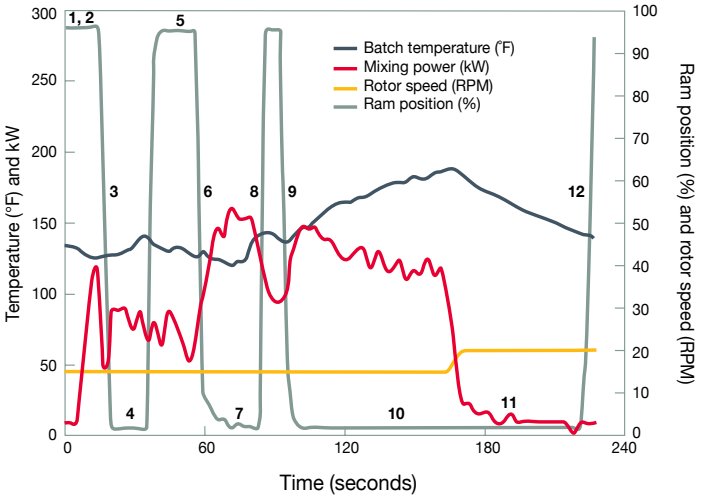


Figure 3: Final batch mixing curves for ADC and OBSH blend formulation

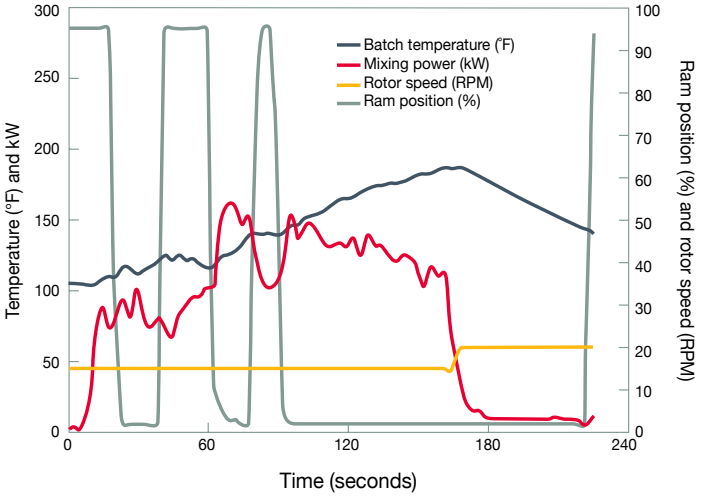


Figure 4: Final batch mixing curves for low nitrosamine formulation

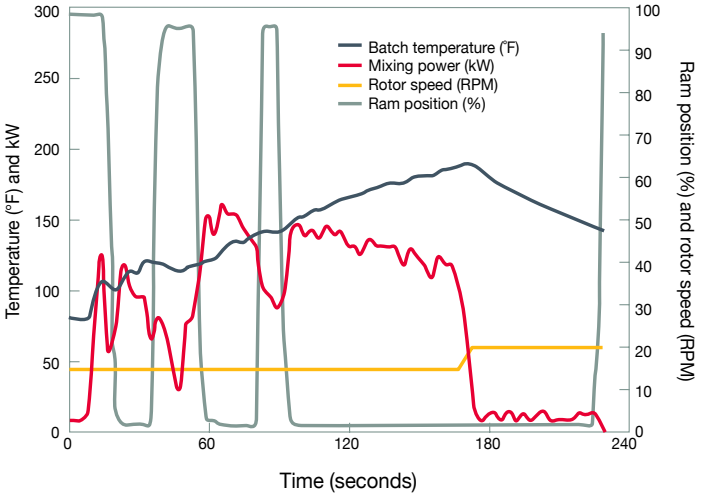
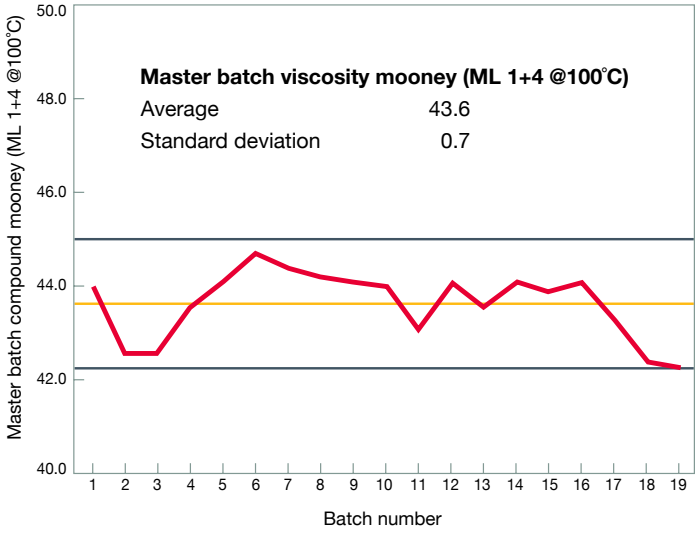


Figure 5: Control chart for master batch compounds



Master batch compound analysis

The model formulations described were utilized to prepare multiple production master batches with consistent rheological properties. The average Mooney Viscosity for the model formulations (Figure 5) was 43.6 MU.

Final batch compound analysis

The NORDEL™ 6555 OE EPDM-based sponge compound was designed to have a fast cure rate in order to balance curing and foaming properties during the vulcanization process. The target ts2 at 180°C was approximately 0.4 min. However, the formulation utilizing OBSH as the blowing agent yielded even faster cure rates (ts2 at 180°C <0.4 min). A low nitrosamine cure package was successfully developed to achieve the same target cure rate of 0.4 min (see Table 6).

Table 6: Final batch compound analysis

Test conditions	Properties	Units	100% ADC formulation	ADC + OBSH blend formulation	Low nitrosamine formulation
ML @ 275°F (135°C)	ML	dNm	32.10	32.40	32.10
	ts5	min	**2.53**	**2.17**	**2.46**
MDR @ 180°C and 8 min	ts1	min	0.35	0.33	0.32
	ts2	min	**0.44**	**0.39**	**0.41**
	t10	min	0.39	0.36	0.36
	t50	min	**1.27**	**0.85**	**1.4**
	t90	min	**4.29**	**3.22**	**4.63**
	ML	dNm	**1.17**	**1.20**	**1.22**
	MH	dNm	15.78	15.86	15.73
MDR @ 160°C and 8 min	ts1	min	0.66	0.6	0.59
	ts2	min	**0.89**	**0.77**	**0.82**
	t10	min	0.74	0.67	0.64
	t50	min	**2.39**	**1.74**	**2.59**
	t90	min	**5.24**	**5.13**	**5.73**
	ML	dNm	**1.32**	**1.35**	**1.35**
	MH	dNm	14.72	14.86	13.79

Extrusion and vulcanization

NORDEL™ 6555 OE EPDM-based sponge compounds were extruded on a 3.5-inch rubber cold feed extruder with a 10:1 L/D ratio to form the desired profile shape shown in Figure 6.

The extruded profile was continuously cured on a continuous vulcanization (CV) line, which is a combination of multiple hot air and microwave ovens. High quality, closed cell sponge profiles were successfully produced via the previously mentioned process. In this particular study, the CV line consists of a total of three 20-foot ovens (see Table 7). The first and third ovens are hot air ovens with adjustable speed, temperature, and air velocity. The second oven is a 20-foot microwave oven with three adjustable power outputs (max power 6 kW) as well as adjustable speed, temperature, and air velocity.

Figure 6: Omega testing profile die utilized for extrusion

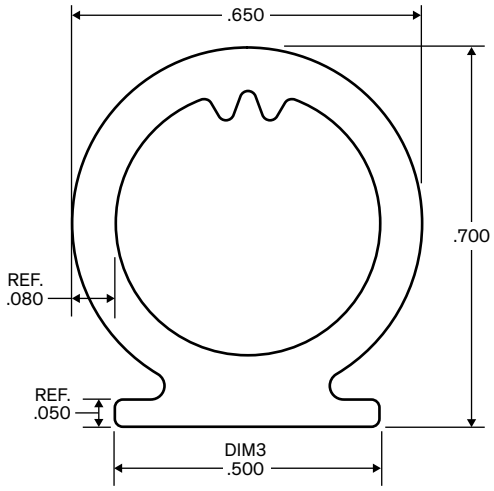


Table 7: Extrusion and vulcanization

Extruder	Units	100% ADC formulation	ADC + OBSH blend formulation	Low nitrosamine formulation
Screw temperature	°F (°C)	125 (52)	125 (52)	125 (52)
Zone 1 temperature	°F (°C)	150 (66)	150 (66)	150 (66)
Zone 2 temperature	°F (°C)	150 (66)	150 (66)	150 (66)
Zone 3 temperature	°F (°C)	150 (66)	150 (66)	150 (66)
Zone 4 temperature	°F (°C)	150 (66)	150 (66)	150 (66)
Speed	RPM	18	18	18
Hot air oven #1				
Oven length	ft	20	20	20
Temperature	°F (°C)	430 (221)	430 (221)	430 (221)
Speed	fpm	30	30	30
Microwave oven				
Oven length	ft	20	20	20
Temperature	°F (°C)	480 (249)	450 (232)	480 (249)
1-bottom	kW	1.5	0.8	1.7
2-top	kW	1.4	1.0	1.7
3-bottom	kW	1.5	1.0	1.7
Speed	fpm	33	35	33
Hot air oven #2				
Oven length	ft	20	20	20
Temperature	°F (°C)	500 (260)	500 (260)	500 (260)
Speed	fpm	37.3	38.5	39.1
Last belt				
Speed	fpm	40.3	41.2	40

EPDM sponge profile properties

Good surface quality and uniform closed cell structures were achieved for all three model formulations. Nevertheless, differences in cell morphology were observed across formulations. Most significantly, sponge profiles generated using the ADC/OBSH blend formulation had more cell rupture, more opened cells, and skin that was less dense relative to the 100% ADC and low nitrosamine formulations.

Figure 7: Cross-sectional morphology of EPDM sponge profile based on 100% ADC formulation

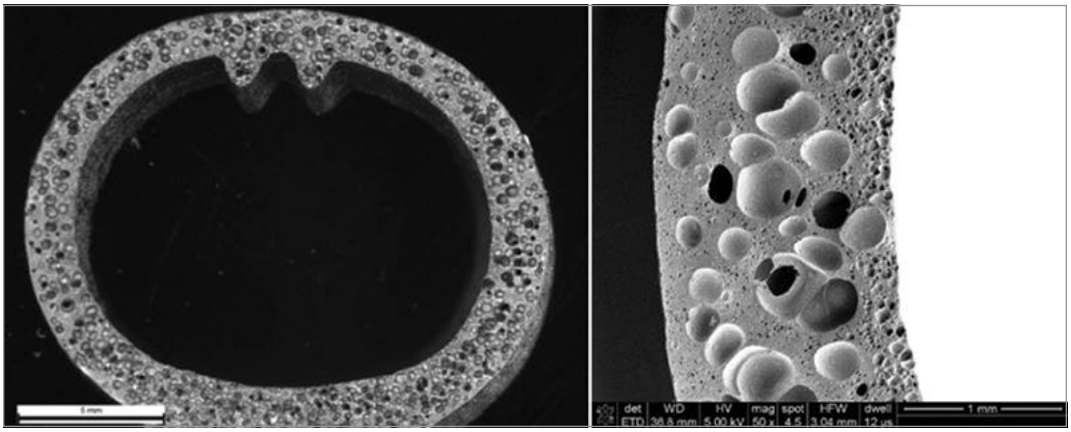


Figure 8: Cross-sectional morphology of EPDM sponge profile based on ADC + OBSH blend formulation

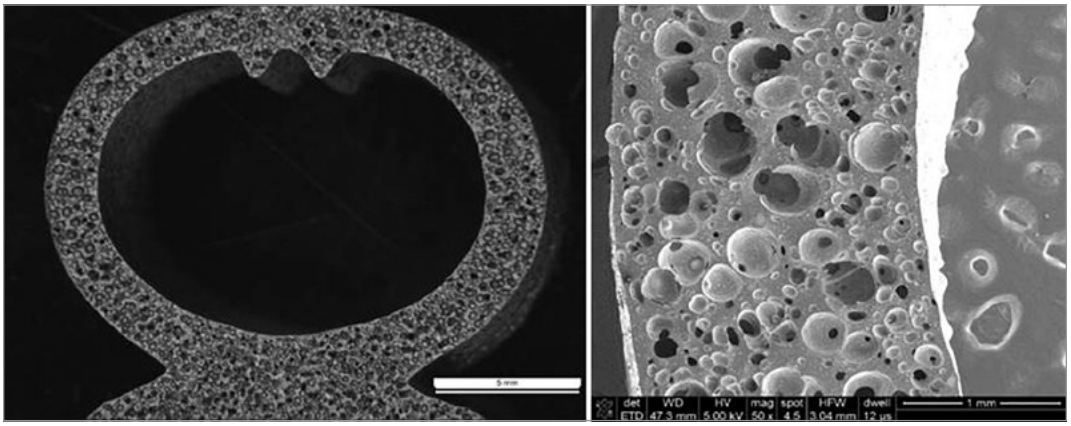
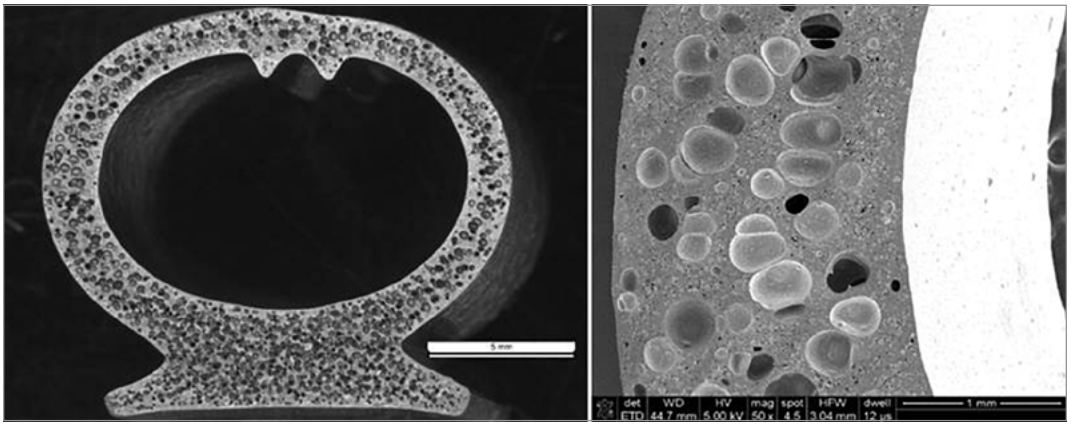


Figure 9: Cross-sectional morphology of EPDM sponge profile based on low nitrosamine formulation



All model formulations yielded sponge profiles with low water absorption, low compression set and good surface quality at target densities (see Table 8). Additionally, the tensile and tear properties exceeded specification targets.

Table 8: EPDM sponge profile properties[1]

Properties[2]	Units	Specification[3] VW-TL52704 and Chrysler-MS-AK-92/Sponge grade 2	100% ADC formulation	ADC + OBSH blend formulation	Low nitrosamine formulation
Density	pcf (g/cc)	28-41 (0.45-0.65)	36.7 (0.59)	36.4 (0.58)	36.4 (0.58)
CLD[4]	lbf (kgf)	–	0.8 (0.36)	1.6 (0.64)	1.6 (0.64)
CLD[5]	lbf (kgf)	–	1.4 (0.64)	2.4 (1.09)	2.7 (1.22)
Compression set[6]	%	–	2.2	2.4	2.8
Compression set[7]	%	–	17.5	19.0	18.1
Water absorption	%	≤5	0.50	3.50	0.15
Tensile strength	psi (MPa)	≥435 (≥3)	587.6 (4.1)	487.0 (3.4)	645.4 (4.4)
Tear (Die °C) strength	lbf/in (kN/m)	≥11.4 (≥2.0)	58.5 (10.2)	59.0 (10.3)	63.4 (11.1)

[1] Data per tests conducted by Dow. Additional information available upon request. Properties shown are typical, not to be construed as specifications. Users should confirm results by their own tests.
[2] Measurements were taken on fabricated sponge profiles.
[3] This term and any corresponding data refers to typical performance in the specific tests indicated and should not be construed to imply these materials meet VW-TL52704 and Chrysler-MS-AK-92/Sponge Grade 2 sponge specifications.
[4] Compression Load Deflection: Compress 25% on a 100 mm profile. The profile was pre-flexed three times at 40% deflection.
[5] Compression Load Deflection: Compress 40% on a 100 mm profile. The profile was pre-flexed three times at 40% deflection.
[6] Compression Set: Compress 50% deflection for 22 hours at room temperature on 100 mm piece and recovery for 24 hours.
[7] Compression Set: Compress 40% deflection for 22 hours at 70°C on 100 mm piece and recovery for 2 hours.

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