PoleStar 200R Calcined China Clay vs Siliceous Kaolins

An Evaluation in an EPDM Extrusion Formulation

Summary

1. PoleStar 200R gives a better balance of extrusion properties than Neuburg Silicas when tested in both soft and hard EPDM extrusion formulations.
2. PoleStar 200R gives a harder compound than the Neuburg Silicas.
3. PoleStar 200R matches the best of the Neuburg Silicas in compression set performance.
4. Ageing at 120°C has a greater effect on compounds containing PoleStar 200R than on those containing Neuburg Silicas.
5. PoleStar 200R gives significantly whiter EPDM compounds than Neuburg Silicas.
6. Compounds containing PoleStar 200R have better dimensional stability than those containing Neuburg Silicas.
7. Subjectively, PoleStar 200R gives better scratch resistance than Neuburg Silicas.

Introduction

The Sillitin range of siliceous kaolins from Hoffman Mineral are widely used in the production of EPDM extruded seals for the automotive industry.

Siliceous kaolins are a natural combination of corpuscular quartz and lamellar kaolinite, which are claimed to give a hard, scratch resistant surface with good extrudability in EPDM.

Previous work[1] showed that **PoleStar 200R** performed well against Sillitins in this application, and so the present evaluation was carried out to confirm the initial results.

Experimental

Samples of Sillitins N85, Z86 and Sillikolloid P87 were obtained and sent for physical, chemical and mineralogical analyses to compare their properties with those of **PoleStar 200R.**

An EPDM masterbatch was prepared in a Banbury internal mixer, and the filler:oil blend was incorporated into the masterbatch using a Twin Roll Mill. The formulation is shown below.

	phr
Keltan 778 EPDM	100
Zinc Oxide	5
Stearic Acid	1
Kezadol GR	10
Paraffin Wax	3
Perkadox 1440	7
EDMA	1
Polyvest 25	2
Sunpar 2280	70 and 100
Filler	200

The two filler:oil ratios (Formulation 1 - 200:70, Formulation 2 - 200:100) were used to demonstrate the effects of the fillers in relatively hard and soft formulations respectively.

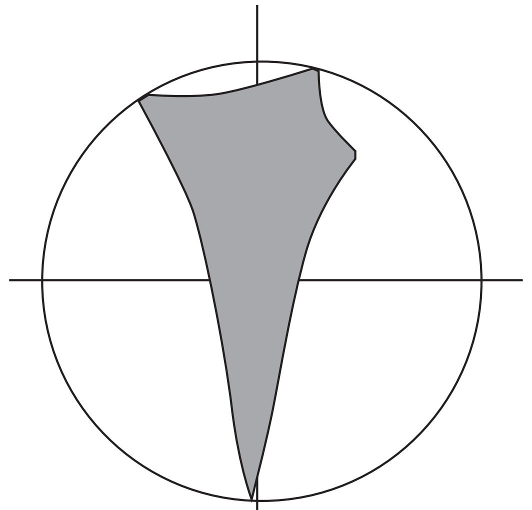
Sample plaques of each compound were cured for 20 minutes at 180°C, and then left to condition for 5 days at 23°C before testing for colour, tensile strength, modulus, elongation, tear strength, hardness and scratch resistance. Tensile testing was also carried out on compound that had been aged for 7 days at 120°C. Compression set test pieces were moulded under the same conditions and tested for percentage recovery after 24 hours at 120°C.

The curing characteristics at 180°C were measured on both formulations using a Monsanto Oscillating Disc Rheometer.

Each compound was also tested for extrudability, according to ASTM D 2230, with a single screw extruder fitted with a Garvey Die. The Garvey Die extrusion profile and appearance rating system are illustrated and described below (Figures 1 and 2).

Extruded sections of both formulations were cured in a steam autoclave at 170°C for 20 minutes. Profiles from Formulation 1, which showed the largest shrinkage, were measured to calculate the percentage shrinkage on cure.

Figure 1 - Garvey Die Extrusion Profile - ASTM 2230



This profile is specially shaped to produce an extrusion with a combination of relatively flat surfaces, sharp corners and thin sections. The test compounds are extruded through the die following standard conditions (110°C and 45rpm), and the nature of the extrusion is rated visually for smoothness, sharp corners, and integrity of thin sections according to the system shown in Figure 2.

[1] A Comparison of **PoleStar 200R** with Sillitin Z86 in Light Coloured EPDM Extrusions, *D.A. Skelhorn & D.J. Bray*

This rating system is based on four separate gradings, each grading represented by a digit. The first digit in the rating refers to swelling or porosity, the second digit to sharpness and continuity of the 30° edge, the third digit refers to smoothness of the surface, and the fourth digit refers to sharpness and continuity of corners. The ratings range from 1 (poor) to 4 (excellent), and the four digits are added to give a total rating out of 16.

Results and Discussion

Filler Analysis - Table 1

A comparative analysis of the fillers shows that the only significant variation in the three Hoffmann minerals is their particle size distributions. The Sillitin N85 has a higher +10µm content than Sillitin Z86 and Sillikolloid P87, which are both similar in value. **PoleStar 200R** has a slightly higher +10µm content than the Hoffmann minerals, but it is equivalent in coarse particle (+53µm) content. The largest difference in particle size is seen at the -2µm level where both **PoleStar 200R** and Sillitin N85 are the coarsest at 52-53 wt%. Sillitin Z86 is finer with a -2µm content of approximately 70 wt%, and Sillikolloid in much finer again with about 87 wt% -2µm. Chemical and mineralogical analysis shows that the three Hoffmann minerals are very similar, with the high quartz content that is characteristic of Neu7burg Silicas. **PoleStar 200R** has the typical chemical composition of a calcined English china clay, and is almost totally amorphous.

Cure Testing - Table 2

All four fillers give similar scorch times (T_2) and cure times (T_{90}) when tested in Formulation 1. The minimum torque is slightly lower for Sillitin N85 and the maximum torque is slightly lower with Sillitin Z86. In Formulation 2, the scorch times are equivalent, but the cure times for Sillitin Z86 and Sillikolloid P87 compounds are approximately half that of **PoleStar 200R** and Sillitin N85 compounds. The minimum torque values are all similar, but Sillitin N85 and Sillikolloid P87 compounds give lower maximum torque values. Cure shrinkage results show that **PoleStar 200R** compounds have a significantly higher dimensional stability than those with the Neuburg Silicas.

Extrudability - Table 3

The Garvey Die ratings in Table 3 show that **PoleStar 200R** gives the best overall extrudability. In Formulations 1 and 2, **PoleStar 200R** gives the highest extrudability ratings of 13 and 14 respectively. Sillikolloid P87 also gives a rating of 14 in Formulation 2, but its surface is not as good as that given by **PoleStar 200R.** Samples of the extrudate are shown in Figure 3.

Tensile Testing - Table 4

In both formulations, the results follow a similar pattern with the Sillikolloid P87 giving the highest peak strength, lowest elongation and the stiffest compound, which would be expected from its finer particle size. The remaining three fillers give compounds with generally similar tensile properties.

Aged Tensile Testing - Table 5

Ageing of all the compounds results in a modulus increase and a corresponding fall in elongation at break. Formulation 2 is relatively unaffected by ageing due to its higher oil content, but in Formulation 1 the **PoleStar 200R** filled compound gave the worst ageing, which may be due to a cross-link density effect on curing, but this needs investigation.

Physical Testing - Table 6

Hardness - Two methods were used to test hardness because of its importance in relation to scratch resistance. Neuburg Silicas are often used in EPDM extrusions because it is claimed that they give a harder surface and therefore a more scratch resistant compound. This testing shows that **PoleStar 200R** gives the hardest compounds on consideration of both formulations and both test methods.

- **Tear Strength** - Crescent tear strength results are similar for all compounds in both formulations.
- **Compression Set** - In the harder, low oil Formulation 1, the best percentage recovery properties are given by **PoleStar 200R** and Sillitin N85. The Sillikolloid P87 gives about 5% less recovery, and Sillitin Z86 is significantly worse, giving about 10% less recovery.
- **Colour** - In both formulations **PoleStar 200R** gives a significantly whiter compound than the Neuburg Silicas, which all give similarly coloured compounds.
- **Scratch Resistance** - Our standard scratch test was applied to moulded sheets of the compounds to try and measure scratch resistance. The Sillitin N85, Z86 and Sillikolloid P87 compounds were visibly much less scratch resistant than the **PoleStar 200R** compound, but it was impossible to quantify the differences because of colour variation between compounds and the elastic behaviour of the rubber surface.

Figure 2 - Garvey Die Appearance Rating System

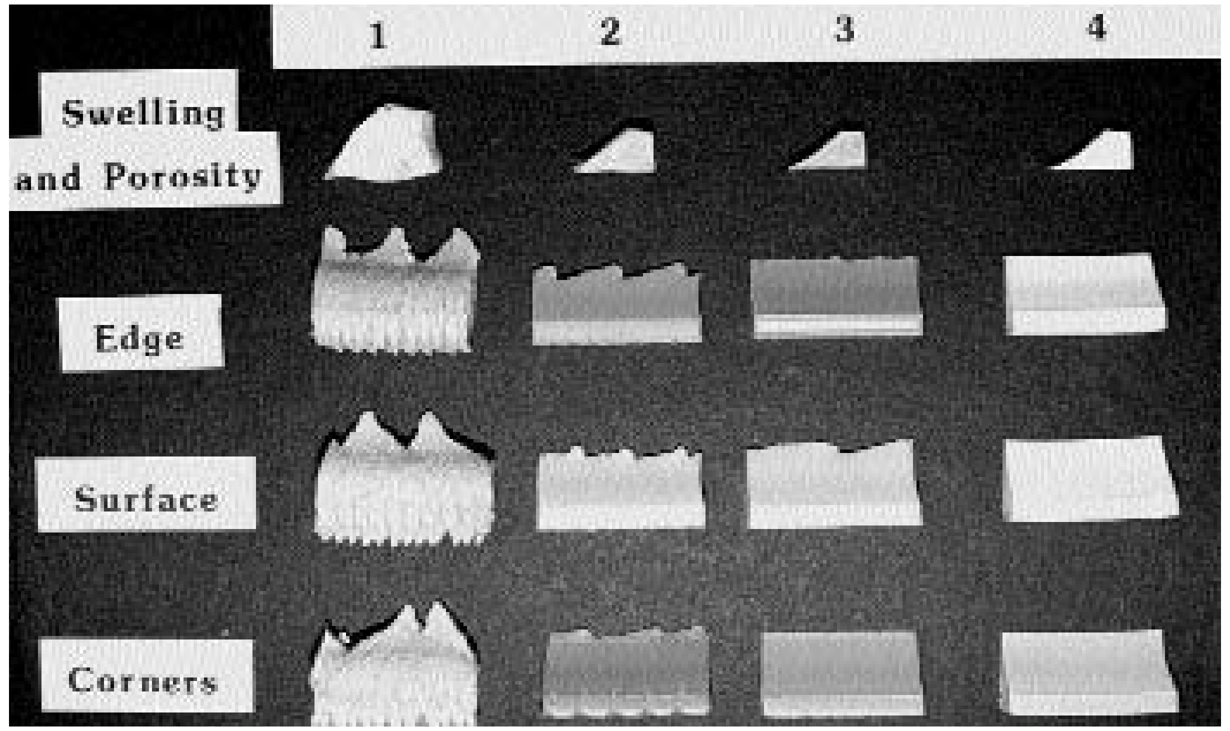


Figure 3 - EPDM Garvey Die Extrusions

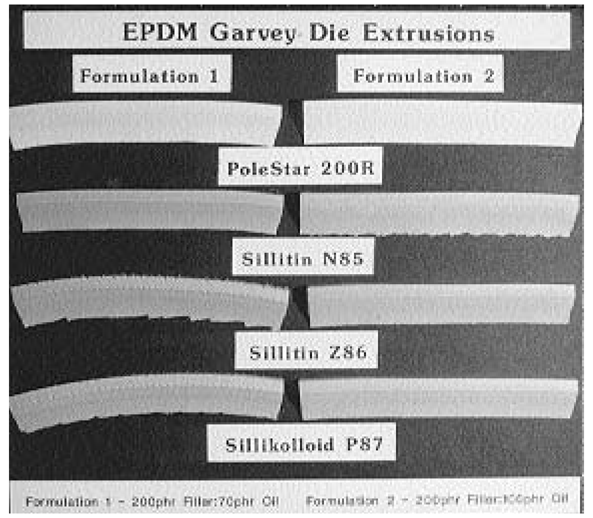


Table 1 - Filler Analysis (Typical Values)

Property	PoleStar 200R	Sillitin Z86	Sillitin N85	Sillikolloid P87
Particle Size				
ppm + 53µm	110	100	100	100
wt% +10µm	12.0	1.3	2.5	0.8
wt% -2µm	52.5	69.5	52.1	86.5
XRF(wt%)				
SiO_2	55.0	82.6	85.5	77.6
Al_2O_3	42.0	12.0	9.8	15.3
Fe_2O_3	0.50	0.61	0.59	0.77
TiO_2	0.02	0.14	0.16	0.17
CaO	0.01	0.04	0.04	0.06
MgO	0.17	0.07	0.09	0.09
K_2O	1.77	0.36	0.31	0.43
Na_2O	0.04	0.01	0.01	0.01
LOI	0.2	4.2	3.6	5.6
XRD (wt%)				
Kaolinite	-	25	17	26
Mica	-	2	1	1
Quartz	1	71	77	65

NB. The quartz total consists of crystalline quartz and X-ray amorphous microcrystalline silica.

Table 2 - Curing Characteristics

Filler	Formulation 1					Formulation 2			
	Max Torque (lb/in)	Min Torque (lb/in)	T2 (min)	T90 (min)	Shrinkage (%)	Max Torque (lb/in)	Min Torque (lb/in)	T2 (min)	T90 (min)
PoleStar 200R	42	13	2	7	5	19	4	2	14
Sillitin N85	36	8	2	8	9	20	5	2	14
Sillitin Z86	34	12	2	8	10	12	5	2	6
Sillikolloid P87	38	13	2	7	9	13	5	2	7

Table 3 - Extrudability Ratings

Filler	Formulation 1 Garvey Die Rating	Formulation 2 Garvey Die Rating
PoleStar 200R	13	14
Sillitin N85	11	11
Sillitin Z86	8	13
Sillikolloid P87	10	14

Table 4 - Tensile Results

Filler	Formulation 1				Formulation 2			
	Peak Strength (MPa)	Elong at break (%)	Modulus 100% (MPa)	Modulus 300% (MPa)	Peak Strength (MPa)	Elong at break (%)	Modulus 100% (MPa)	Modulus 300% (MPa)
PoleStar 200R	7.3	376	2.9	5.9	5.3	771	1.2	3.2
Sillitin N85	6.5	516	2.0	4.9	5.2	804	1.0	2.7
Sillitin Z86	5.3	412	2.1	4.9	6.1	756	1.2	3.1
Sillikolloid P87	8.4	388	2.7	6.5	7.4	673	1.5	3.9

Table 5 - Aged Tensile Testing Results

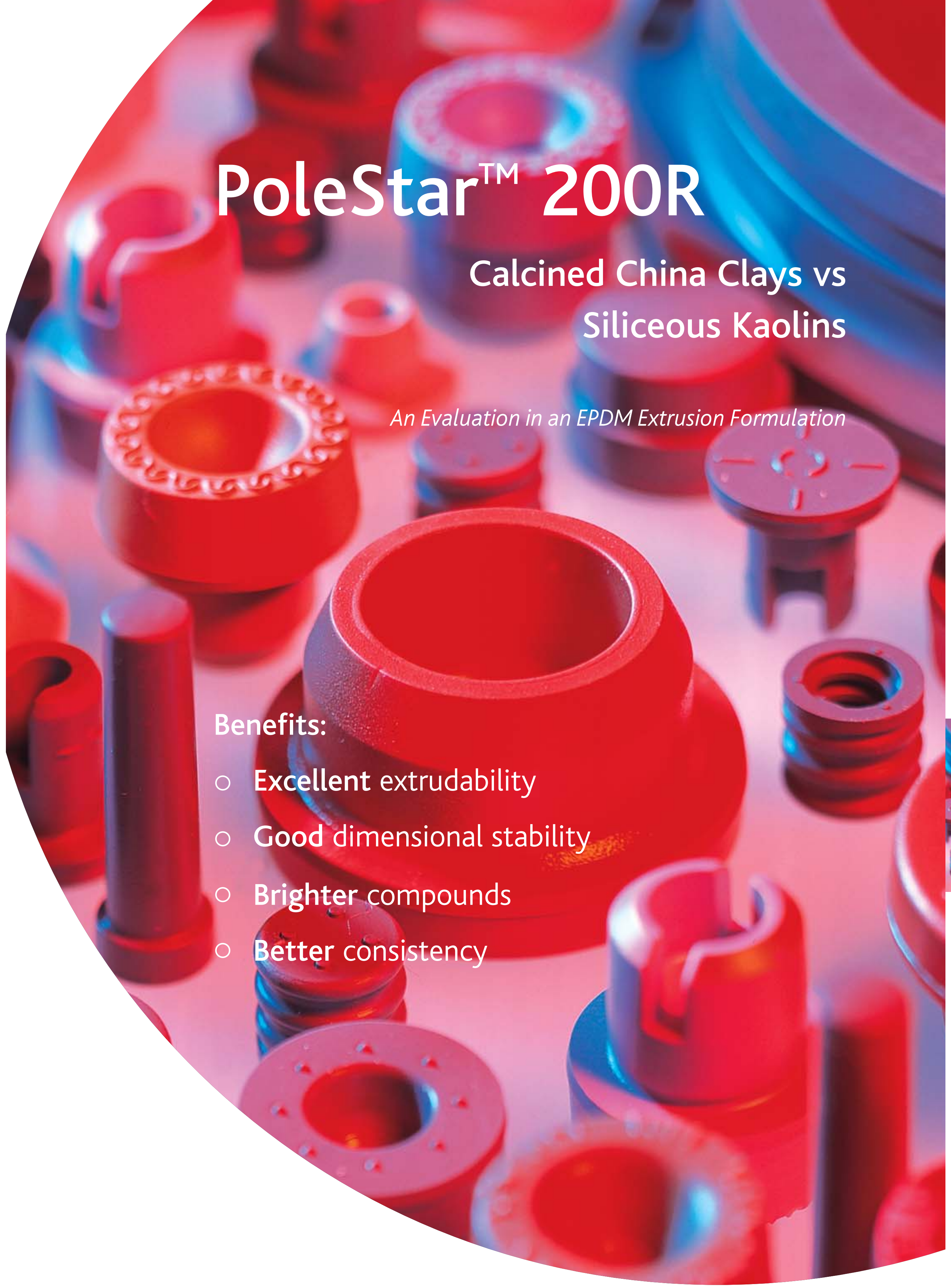
Filler	Formulation 1				Formulation 2			
	Peak Strength (MPa)	Elong at break (%)	Modulus 100% (MPa)	Modulus 300% (MPa)	Peak Strength (MPa)	Elong at break (%)	Modulus 100% (MPa)	Modulus 300% (MPa)
PoleStar 200R	6.2	187	3.9	-	4.4	339	1.6	4.2
Sillitin N85	6.1	363	1.9	5.5	5.3	707	1.0	3.0
Sillitin Z86	6.1	349	2.2	5.7	6.8	703	1.2	3.5
Sillikolloid P87	8.6	308	3.0	8.3	6.7	529	1.4	4.1

Table 6 - Physical Testing

Filler	Formulation 1				Formulation 2			
	IRHD Hardn'ss	Shore A Hardn'ss	Crescent Tear (Nmm)	Comp. Set Recovery (%)	IRHD Hardn'ss	Shore A Hardn'ss	Crescent Tear	Comp. Set Recovery (%)
PoleStar 200R	52.7	68.3	30.7	28.9	41.0	58.0	28.3	34.4
Sillitin N85	48.0	65.3	31.1	31.2	38.7	57.0	28.2	35.5
Sillitin Z86	47.7	66.0	28.4	20.9	40.3	55.3	31.8	33.2
Sillikolloid P87	50.7	69.3	32.4	25.4	41.7	57.0	32.5	35.5

Table 7 - Colour Measurements

Filler	Formulation 1				Formulation 2			
	L	a	b	Delta E	L	a	b	Delta E
PoleStar 200R	82	-0.7	17	25	81	-0.6	18	26
Sillitin N85	63	1.2	29	47	64	1.0	29	46
Sillitin Z86	63	2.1	30	48	65	1.7	29	46
Sillikolloid P87	65	2.0	28	45	65	1.5	28	45



PoleStar™ 200R

Calcined China Clays vs Siliceous Kaolins

An Evaluation in an EPDM Extrusion Formulation

Benefits:

- **Excellent** extrudability
- **Good** dimensional stability
- **Brighter** compounds
- **Better** consistency

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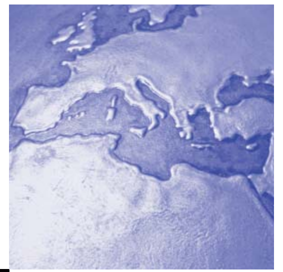

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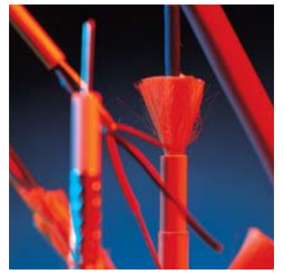

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