

Introduction

Calcium Hydroxide is used to activate the incorporated fluoropolymer cure system in FKM fluoro elastomers. The bisphenol cure system forms a double bond by eliminating hydrofluoric acid from the polymer backbone, which is neutralised by the calcium hydroxide.

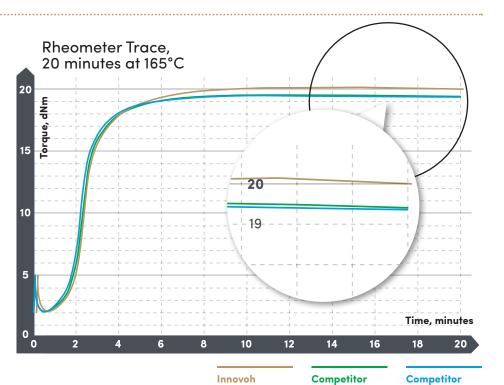
Summary

- Innovoh / (Innovox OH) exhibits a higher state of cure than the competitor material studied, indicating a higher crosslink density.
- This manifested itself in a lower methanol volume swell.
- Innovoh is significantly better dispersed than either competitor.

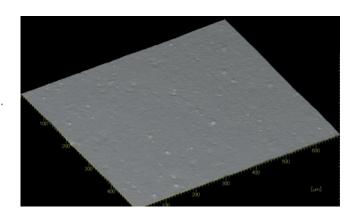
Graph 1

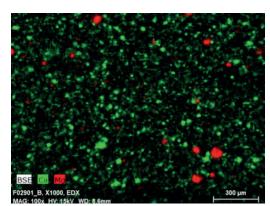
Curing characteristics of Bispenol Cured FKM compound

• The surface roughness (dispersion) evaluated via Scanning Electron Microscopy using a 4 segment backscattered detector showed Innovoh to have the best dispersion. Competitor R and Competitor N showing lower, but similar levels of dispersion to each other.



Innovoh SEM, showing optimum dispersion







Experimental summary

Table 1FKM Masterbatch
using Cures in
Polymer

• The samples were mixed as per the following formulations:

	F002866A	
	FKM Masterbatch	
FKM FC2181	100.0	
Garolite DE	3.0	
Thermax MT	30.0	
Total Parts	133.0	

Table 2Recipe mix and
test designation

• The hydroxides were added to FKM masterbatch on a 2-Roll Mill. Once all the powder was incorporated, the compound was rolled up and passed through a tight nip end-on 5 times, rolling the compound each time. Dispersion was determined by SEM (Scanning Electron Microscope).

Table 3 Test methods

• The compounds were cured for 15 minutes at 165°C and then post cured for 16 hours at 230°C. The masterbatch without additive did not cure and was omitted from any further work. The physical properties of these samples were tested as in table 3 (see opposite).

	F002866A	F002866B	F002866C	F002866D	
	FKM Masterbatch	FKM Innovoh Control	FKM Competitor N	FKM Competitor R	
FKM Masterbatch	133.0	133.0	133.0	133.0	
Innovoh		6.0			
Competitor N			6.0		
Competitor R				6.0	
Total Parts	133.0	139.0	139.0	139.0	

Physical Property	Test Method
Hardness	DIN 73411-2
Tensile Strength	ISO 37
Volume Swell	ISO 1817
Compression Set	ISO 815

Results and discussion

- There was noticeably less powder falling onto the tray from the mill on the addition of Innovoh to the rubber compared to the other samples tested.
- Innovoh exhibits a higher state of cure over all samples tested.
 While Competitor R shows faster cure than the other samples tested, this can be considered negligible as FKM is post cured.



Table 4 Rheometer Data

Rheomete	r	Norm	ISO 3417			
Test Tempe	rature [°C]	165		·		
Test Time	[Mins:Sec]	20:00				
		МВ	Innovoh Control	Competitor N	Competitor R	
ML	[lb-in]	1.57	1.99	1.97	2.00	
ts1	[Mins:Sec]	4:03	1:21	1:22	1:17	
ts2	[Mins:Sec]	6:16	1:40	1:41	1:37	
ts50	[Mins:Sec]	10:13	2:18	2:21	2:14	
ts90	[Mins:Sec]	17:37	4:20	3:55	3:43	
МН	[lb-in]	9.69	20.18	19.57	19:49	

Table 5Physical Properties

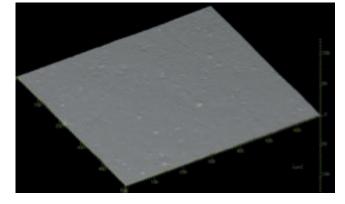
• The physical properties of cured samples are as opposite:

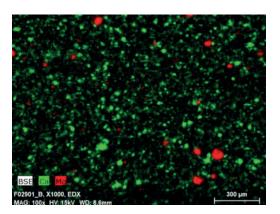
		F02866B		F02866D	
		Innovoh Control	Competitor N	Competitor R	
Hardness	Shore A	74	73	74	
Tensile Strength	MPa	15.0	15.5	15.5	
Modulus at 100%	MPa	5.0	5.0	5.0	
Modulus at 200%	MPa	11.2	10.4	10.5	
Modulus at 300%	MPa		15.5		
Elongation at Break	%	260	301	294	
Aging 70 hours at 200	°C				
Hardness	Shore A	75	74	75	
Tensile Strength	MPa	15.9	16.4	14.5	
Modulus at 100%	MPa	5.4	5.3	5.5	
Modulus at 200%	MPa	12.4	11.7	11.1	
Elongation at Break	%	251	280	248	
Volume Swell					
7 Days at 23°C in Methanol	%	109	115	112	
Compression Set					
70 hours at 200°C	%	19	18	21	



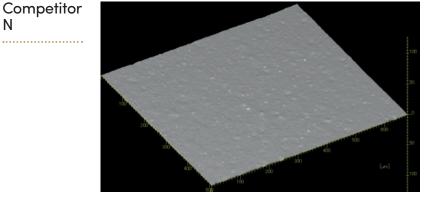
• Innovoh shows the best dispersion when observed under a scanning electron microscope using a 4 segment backscatter detector.

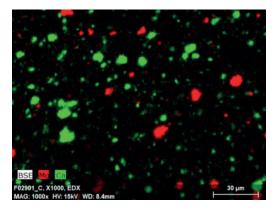
Innovoh Control



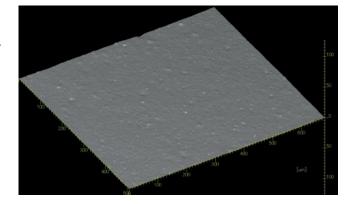


Competitor Ν





Competitor R



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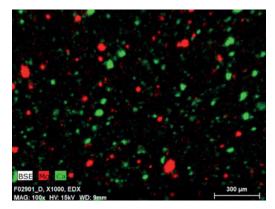




Table 6 Compression Set and Volume Swell		F02866B	F02866C	F02866D	
Methanol volume swell was			Innovoh Control	Competitor N	Competitor R
lower for Innovox OH.	Compression Set: 3 Results	D451132		15.0min. / 165°C	
	Comp. Set	[%]	19.2	17.9	20.6
	Comp. Set	[%]	19.1	18.6	20.1
	Comp. Set	[%]	18.8	17.4	21.4
	Test time	[h]	70	70	70
	Test temp	[°C]	200	200	200
	Test piece	[Ømm]	13	13	13
	Compression Set: 70 hours at 200°C	[%]	19	18	21
	Volume change in liquids	ISO1817		15.0min. / 165°C	
	Volume Swell				
	7 Days at 23°C in Methanol	[%]	109	115	112

Conclusion

All samples performed equally well in terms of physical properties and state of cure. Innovoh gave higher MH on the Rheometer trace indicating a higher crosslinking density. This was confirmed in a lower methanol volume swell. SEM showed the dispersion of Innovoh to be better than the other samples tested. This generally results in better processing, physical properties and so better performance in service.

Tested by Artis Independent Rubber Consultants



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