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CLWYD COMPOUNDERS LTD

Combining Compound Optimization and Mix Cycle Evaluation to Suit Production Techniques and End Product Performance

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Fundamental aspects of compounding high performance elastomers include controlling variability in the mixing process as well as the control and correct selection of ingredients for processes and applications. Manufacturers require reliability, consistency and high output of parts using a variety of moulding techniques. Compounders today must ensure they use available technology to monitor raw materials and mixing parameters to improve the quality of their products.

Introduction

Previous studies on raw material and process control have sought to eliminate batch variation when mixing rubber compounds (Lee, 1999). Additionally we have demonstrated that double mixing high-performance elastomers further reduces batch variation (Febery, 2001). Here we detail the importance of ingredient selection and further processing considerations to achieve consistent high quality rubber compounds tailored to the manufacturer's processes.

Study 1: Process Optimization using Tailored Cure Packages

The aim of this study was to overcome issues related to scorch and increase production rate through the injection moulding process for an EPDM 70 peroxide cured compound.

To achieve this we tailored the cure package using a set of trials identified by design of experiment (DOE) software. To the software we input parameters we can change and the response factors we want to monitor.

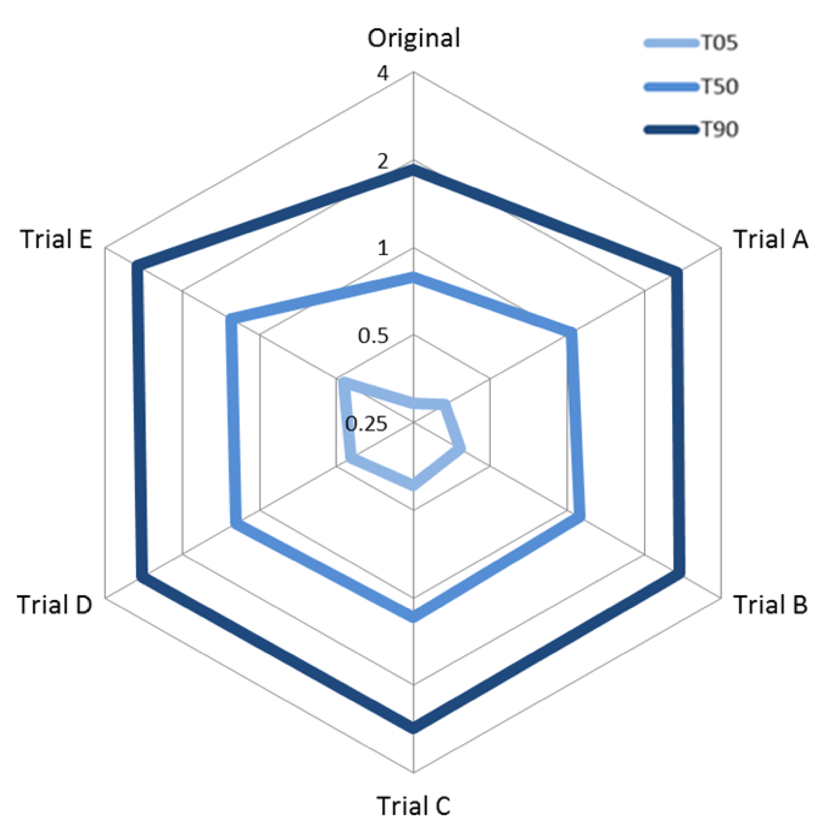

Figure 1: Increase in time to onset of cure

The optimized cure system achieved in Trial E showed increased time to onset of cure; indicated by a longer T05 time. Overall cycle times were not extended; indicated by similar T90 times across the trials. This is a key response factor as it allows more flow time in the mould cavity allowing better mould fill and part quality.

Table 1: Overview of properties following cure system optimisation

		Original	Trial A	Trial E
Basic Properties	ISO 48:2010, ISO 37:2011, BS ISO 34-1:2010			
Hardness	Shore A	66	64	64
Tensile Strength	MPa	13.5	14.7	14.7
Elongation at Break	%	320	379	344
Tear Strength	N/mm	29.3	32.6	32.6
Compression Set	BS ISO 815-1:2008			
24 hours @ 150°C	%	17	15	14
Scorch Time	ASTM 1646			
Scorch @ 121°C	Mins	9.8	18.28	85.21
Rheology @ 185°C	ASTM D5289			
ML	dNm	1.73	1.88	1.68
MH	dNm	21.61	21.48	20.81
T05	Min	0.29	0.33	0.47
T90	Min	1.85	2.67	3.01
Rheology @ 165°C	ASTM D5289			
ML	dNm	1.83	1.98	1.58
MH	dNm	22.23	21.11	19.89
T05	Min	0.32	0.43	0.64
T90	Min	3.9	6.4	6.68

The optimised compound retained the physical properties of the original while showing improved compression set, scorch time and delay to onset of cure. From analysis of the cure properties we found we can safely increase the moulding temperature to reduce overall cycle time without undesired loss of flow or scorch issues.

Conclusions

The optimized compound showed greatly improved processing safety. The improved processing safety allowed the customer to increase the moulding temperature to reduce overall cycle time. Tailoring compounds for customer processes in this way can result in improved efficiency and quality of parts. This optimised compound was used in 3 horizontal injection moulding machines running automatically.

Study 2: Evaluation of Mix Cycles for Further Improvements in Compound Quality

Previous studies conducted show that the double mixing process used as standard at Clwyd, involving a controlled hold time and a refinement step, results in reduced variation between batches. Here we introduce further control on our two stage process by evaluation of mix profiles.

We can monitor power input and temperature during a mix and by analysis we can determine the best order of ingredient addition for different material types and compositions. We can also tailor mix conditions by altering machine parameters such as rotor speed, temperatures and fill factors.

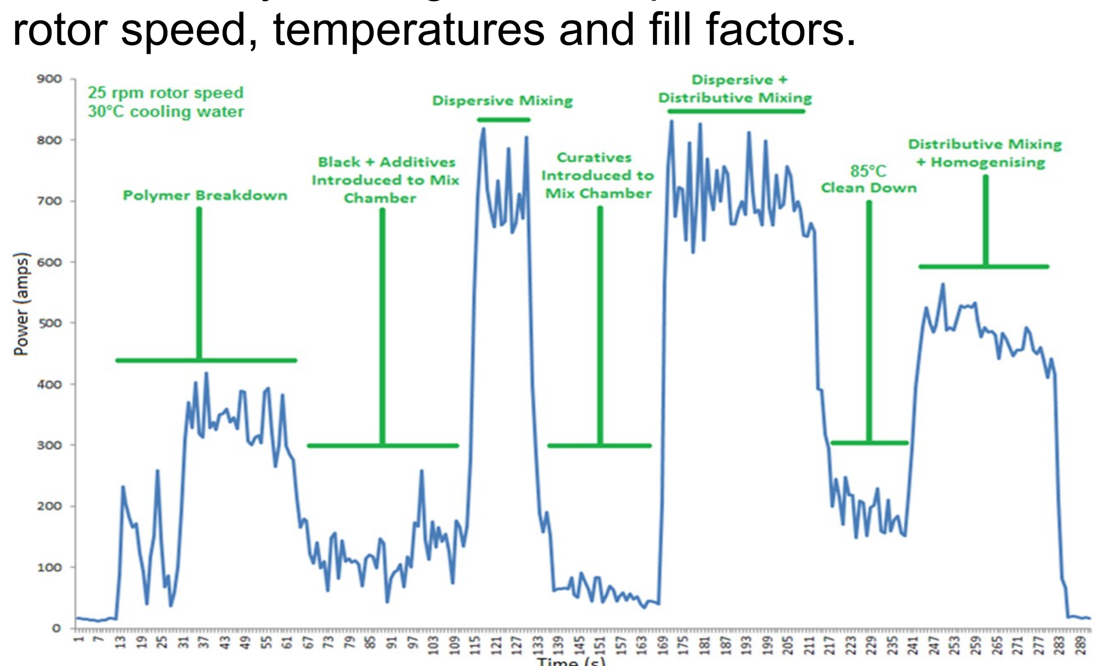

Figure 2: Power chart analysis on 1st stage mix

Correct order and times of addition allow us to maximize dispersive and distributive mixing.

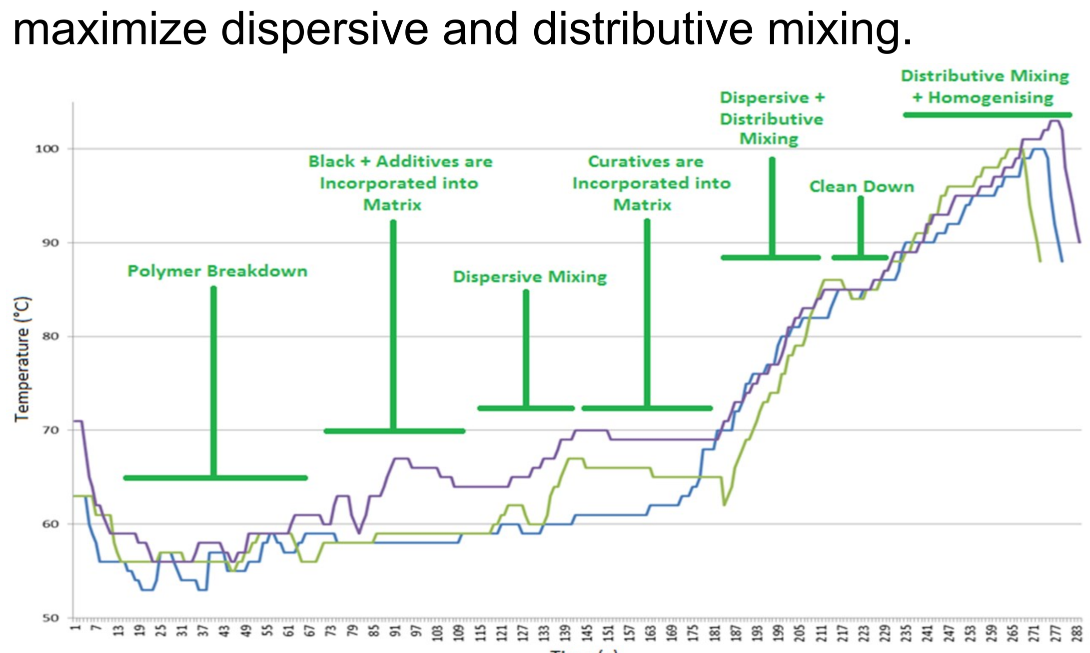

Figure 3: Temperature chart analysis on 1st stage mix

Once a mix sequence has been optimised we can consistently produce compound to a very high quality in an efficient manner.

The 2 stage process, involving a controlled hold and a second stage remill, allows us to refine the compound to further improve dispersion and distribution of ingredients in the compound.

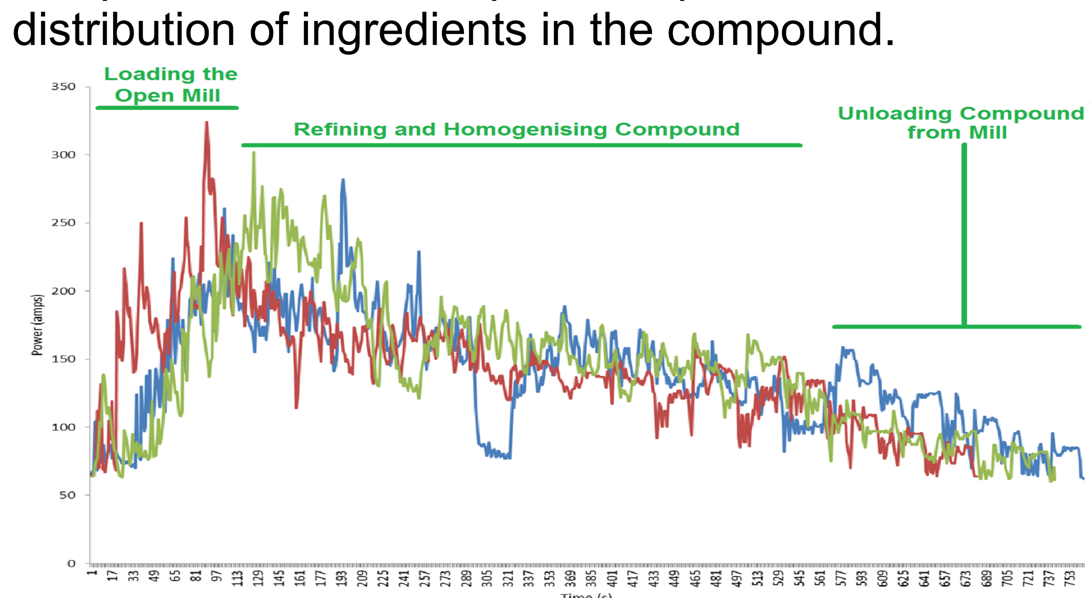

Figure 4: Power chart analysis on 2nd stage remill

The remill stage allows us to achieve tight control on rheology limits ensuring consistent product.

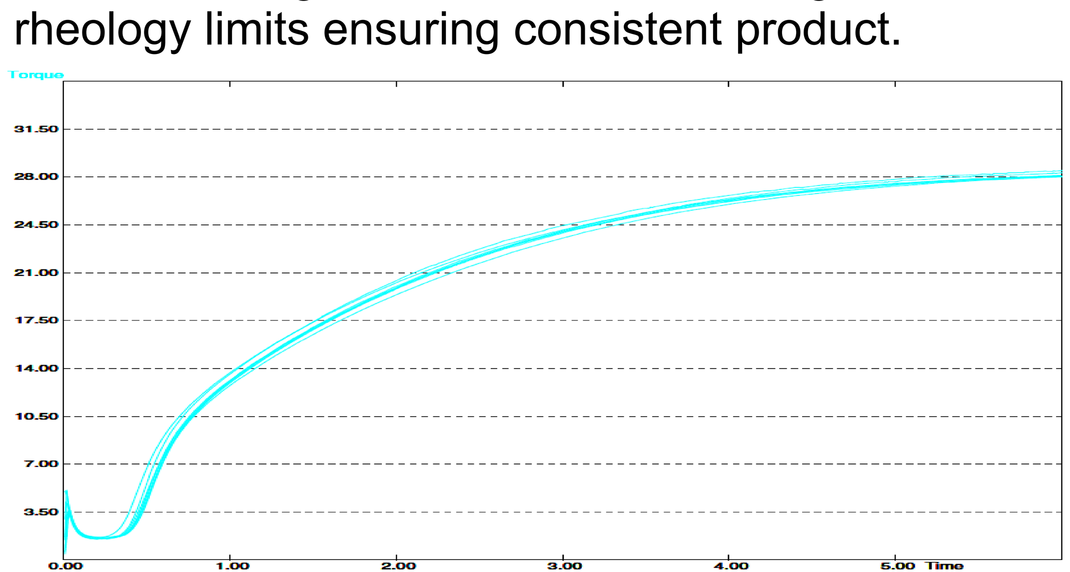

Figure 8: Rheology overlay of a run of HNBR 90 black compound

Conclusions

Enhanced control on mixing parameters allows us to be able to refine mix instructions to ensure optimum dispersion and distribution of ingredients. The 2-stage mixing process allows for enhanced quality of compounds. The outcome of this control and refinement of compounds include:

- 3-sigma control on rheology
- ±1 hardness in a run, ±2 hardness in the year
- Consistent high performance of the compound

Study 3: Developing High Quality Compounds for Extreme Environments

High performance elastomers suited to severe environmental conditions in the oil and gas industry are characteristically tough, high modulus compounds often with high filler loadings. This makes them potentially difficult to mix consistently; which is fundamental to part manufacturers' ability to produce defect free parts. For optimum resistance to extreme temperatures, pressures and aggressive chemicals the quality of a part is paramount.

If a compound is not mixed to a very high standard small particles in the matrix can act as weak points in the structure. These weak points can lead to failure of the part.

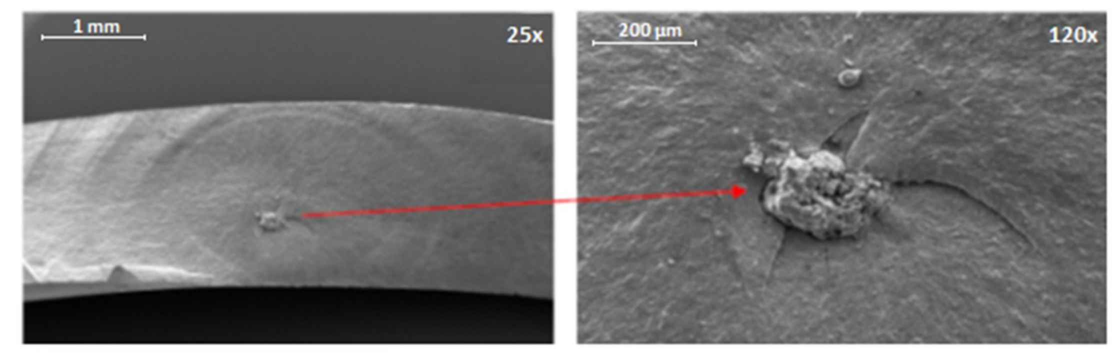

Figure 5: large undispersed particle which lead to failure in testing

When we have control over mix quality, and can ensure reproducibility and reliability, we can supply compounds to handle more extreme conditions.

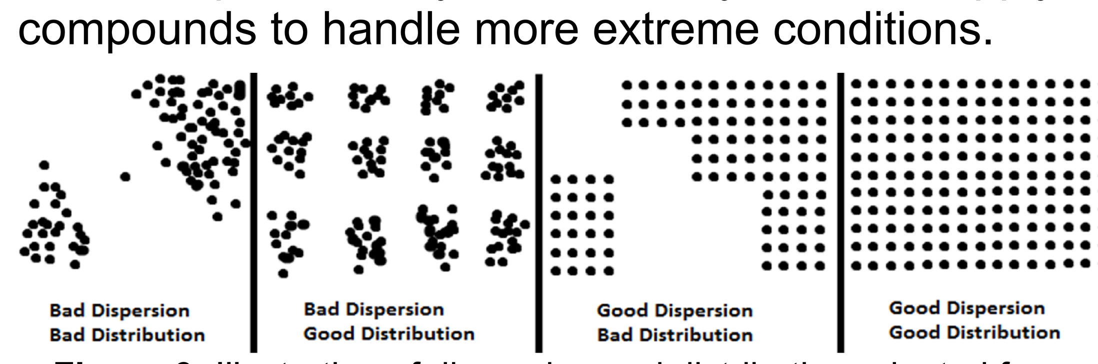

Figure 6: Illustration of dispersion and distribution adapted from www.peptflow.com

Using optimised mix procedures excellent dispersion and distribution of particles and ingredients can be achieved quickly.

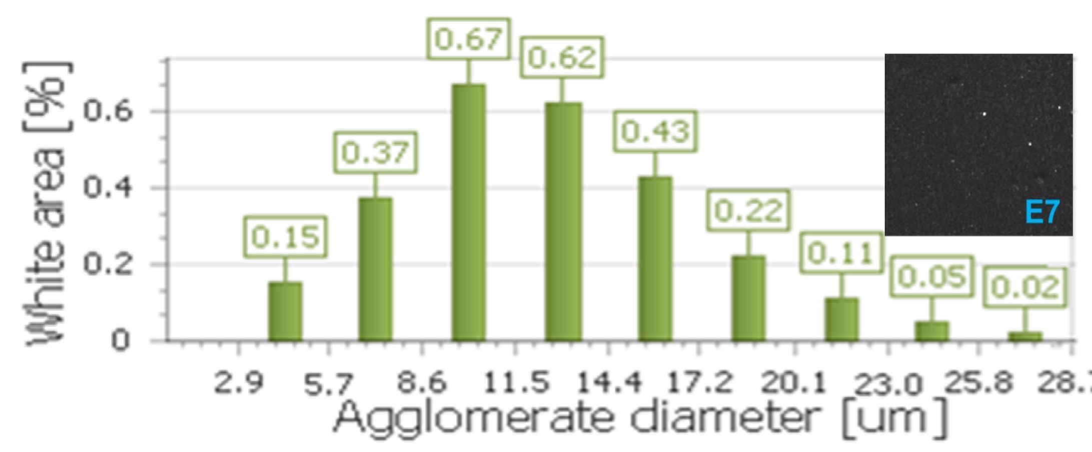

Figure 6: Particle size distribution following 1st stage of mix tested using a dispergrader in accordance with BS ISO 11345

Following a controlled hold to allow structuring and a refinement stage we can achieve ultimate dispersion and distribution of ingredients in the compound.

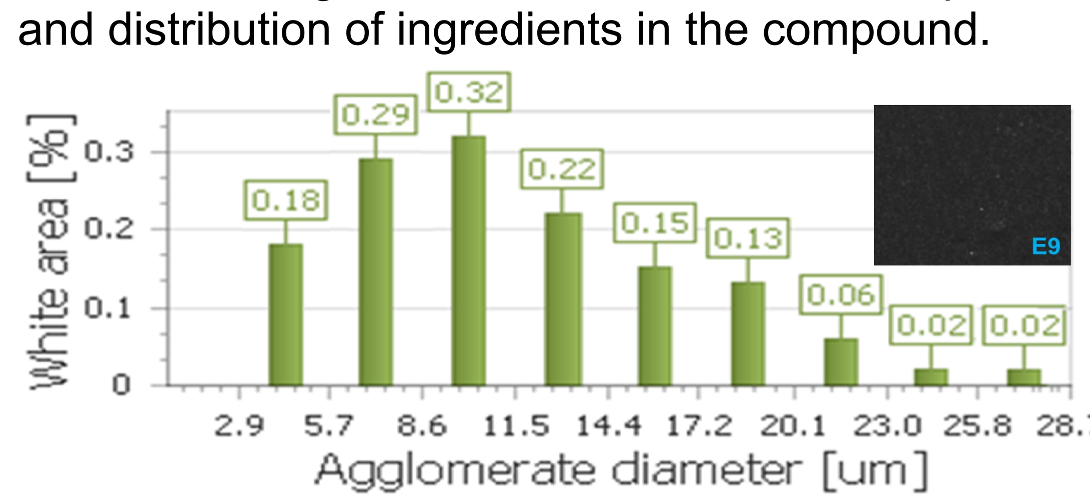

Figure 7: Particle size distribution following 2nd stage remill

The HNBR compound was tested for rapid gas decompression resistance and rated against criteria described in ISO 23936-2. The test pieces showed no signs of cracking and achieved the highest possible rating of 0000.

Table 2: ISO 23936-2 Test Procedure

Gas Composition	90:10 CH_4:CO_2
Temperature (°C)	100
Pressure (MPa)	15
Seal Type	O-ring 2-312

Figure 9: Cut edge of o-ring following EDC testing

Conclusions

With knowledge of compound requirements, use of formulation design and optimised mix procedures we can consistently produce high performance elastomers suitable for extreme environments. Control of mixing parameters and a 2 stage mixing process allows for consistent high quality compound. This allows us to continue to provide high quality elastomer compounds for increasingly demanding conditions in key applications across a range of industries particularly oil and gas.