Keltan® 6251A,

A Versatile Sponge Grade Combining Easy Processing With Excellent Physical Properties.

by

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ABSTRACT

Demands placed on the cost, performance and aesthetics of automotive sponge seals over the past decade have steadily increased. Most automotive sponge formulations use medium to high molecular weight EPDM polymers that must be dispersed in low viscosity compounds designed for low door closing efforts. Efficient and economic mixing of these low viscosity automotive sponge formulations is relatively difficult to achieve. Long mixing times are often necessary to achieve the high quality surface finish of extruded profiles and in many cases a 2-step mixing process is still required to attain expected dispersion levels and to minimize scrap. Due to the extreme demands placed on the cost, performance and quality of automotive sponge, suppliers have little opportunity for improvement. For this reason, Lanxess Elastomers recently embarked on the design, development and commercialization of a new amorphous, high molecular weight, high ENB sponge grade, Keltan® 6251A. The high molecular weight provides excellent physical properties and collapse resistance while the high level of ENB allows ultra fast vulcanization. The incorporation of controlled long chain branching (CLCB)[1] in the polymer microstructure provides an optimal balance of processing and physical properties. With 13 wt% of extender oil, Keltan® 6251A facilitates improved dispersion when compared to polymers of similar molecular weight, providing the opportunity for more economic single stage mixing combined with high quality surface finish. Versus polymers of lower molecular weight, Keltan® 6251A provides the opportunity to reduce cost by extending total loading without sacrificing properties or quality. In this paper it will be demonstrated that Keltan® 6251A is a highly versatile and flexible polymer that can offer significant improvement in mixing performance, cost and/or surface quality, when compared against typical sponge grades and recipes utilized in the market today.

INTRODUCTION

The manufacture of an automotive weatherstrip is a complex and sometimes difficult task. A profile can typically contain anywhere from one to four rubber compounds varying in viscosity, hardness and colour, and may use a supporting metal carrier that all must be co-extruded and cured simultaneously at high speed while meeting tight dimensional tolerances. Of the entire construction, the sponge bulb section of the profile is considered to be the most critical and challenging to manufacture. In a closed position, a car door compresses the sponge bulb providing a seal for the car, in order to prevent air (wind noise) or water entry and to minimize noise within the passenger compartment. Sponge profiles generally utilize a relatively low viscosity EPDM compound that will allow the incorporated blowing agent to generate sufficient gas pressure to expand the surrounding rubber matrix and create a sponge or cellular product. A cellular construction of course reduces the amount of effort it takes to close a door against the bulb section of the profile. However, the viscosity of the sponge compound cannot be too low as it must demonstrate sufficient strength to prevent collapse of the bulb while being processed at high speeds in an unsupported curing environment. At the same time, the sponge formula must possess sufficient cured properties to meet stringent performance requirements demanded by OEM’s. Requirements of good collapse resistance and excellent physical properties usually dictate that a medium to high molecular weight polymer must be dispersed in the low viscosity matrix. Since the surface of the profile must also be smooth and free of any blemishes, undispersed ingredients or foreign matter, efficient and economic mixing of these low viscosity automotive sponge formulations is relatively difficult to achieve. Long mixing times are often necessary to attain the high quality surface finish of extruded profiles and in many cases a two-step mixing process is still required to reach expected dispersion levels and to minimize scrap.

These requirements are not new and have been in place for many years. However, like many automotive parts, the demands for improvement in cost, performance and aesthetics of an automotive sponge seal continue to increase. Over the past several years, manufacturers of automotive profiles have begun to struggle in finding solutions to these ever increasing demands. Opportunities for improvement have diminished as manufacturers reach the limit of materials and processes, and as resources have dwindled due to economic times.

Recognizing this fact, three years ago Lanxess Elastomers decided to embark on the development of a new sponge EPDM polymer that would offer new solutions for their customers. What evolved from the endeavour was much more than just a new polymer. Through the use of a customer survey, several recurring areas for improvement were identified along with the typical dilemmas faced by customers and trends in polymer selection and compound design. This then provided significant input into what polymer characteristics would be needed for the new grade. Based on this, a new sponge grade, Keltan® 6251A was designed, developed and commercialized. This paper will demonstrate that Keltan® 6251A is a highly versatile and flexible polymer that can offer significant improvement in mixing performance, cost and/or surface quality, when compared against typical sponge grades and recipes utilized in the market today. It will also retrace the steps taken in designing the grade, explaining the reasons behind each polymer characteristic selected and how customer dilemmas can be overcome.

Sponge Grade Demands The Past Decade

In the late 90s to early 2000s timeframe, the sponge sealing market was a dynamic environment. At this time, the demands for annual cost reduction programs had already been ingrained in the market and were providing driving forces for sponge development. However, the path to that goal differed by region and was also complimented with other emerging trends. In Japan, OEMs were demanding improvements in surface appearance and cost improvement through weight reduction and increased compound loading. This was mostly accommodated by design changes and the use of lower specific gravity formulations that utilized polymers with higher molecular weight (Mw). In Europe, cost reduction was also attained by specific gravity reduction but with the caveat of performance improvement due to high driving speeds. This required profiles that exhibited good recovery after deformation, improved compression set and smooth surfaces. This drove the use of lower modulus compounds that had extremely fast cure speed. Use of higher Mw, long chain branched grades began to permeate the market. In North America, cost reduction through increased compound loading had been common practice for both sponge as well as solid formulations. However, the need for quality improvement at North American OEMs as well as the influx of European and Japanese car makers forced sponge manufacturers to begin emulating their global counterparts. Compounds utilizing EPDM polymers with higher Mw and unsaturation also became more common place[2].

The same time period saw the introduction of improved mixing equipment and processes. For example, Tier 1 suppliers began installing new intermeshing mixers that had reported superior mixing capabilities, especially for low viscosity recipes. Improvements in mixing technology, combined with market needs for higher Mw polymers drove the introduction of ultra high Mw grades with small amounts of oil extension for best in class profile surface appearance along with superior physical properties. Keltan® 7341A was one such grade[3].

However, the past few years have witnessed much less change and certainly a more stagnant sealing market environment in terms of sponge technology. Regional differences in design and development, although still present, seem to have faded, with many technical discussions dominated by ways to reduce costs. The economic crisis of 2008 added emphasis to this point. Significant downsizing of research staff at OEMs and their entire supply base occurred, adding to an environment of reduced innovation. Financial pressures forced many seal manufacturers to delay or shelve plans to purchase new state of the art mixing equipment or even refurbish existing machines. With little innovation in design, materials and equipment, sponge manufacturers have been forced to continue to repeat and extend old solutions for improvement with diminishing returns as the limit of materials and processes are reached and technical roadblocks are encountered. Also, the last few years have seen the Chinese automotive market emerge along with manufacturers that expect to meet current global standards for cost, performance and quality but with different thought processes and capabilities in manufacturing.

Customer Demands Today

A reduced environment of innovation; less technical staff to make changes; more varied sponge processes; fiscal constraints preventing mixing equipment upgrades and repair; and a strong focus on overcoming "old" cost reduction roadblocks has made new product introductions to the sponge sealing market quite difficult. Of course, since EPDM usually comprises 25-35 weight percent of an automotive sponge formulation, Tier 1 manufacturers still look to EPDM suppliers for help. Recognizing this, three years ago Lanxess Elastomers accepted this challenge and began to ponder how a new grade should be designed. As a first step, a market survey was created and issued to customers globally. Key sponge criteria were categorized into five areas; polymer, formulation, compound mixing, curing and finishing. Customers were then asked to rank existing Lanxess sponge grades against competition in their ability to meet the criteria both today and in the future. Although customers would not divulge competitive grades used, they were willing to identify basic characteristics of the grades. The survey answers provided some enlightening results.

In regard to customer demands; the survey revealed what was just stated. They were looking for cost improvements. Although there were a wide variety of responses, a large number of customers were looking for cost improvements via some aspect of mixing improvement. Some customers wanted to extend their formulations but had trouble obtaining good dispersion. Other customers didn't want formulation change but requested scrap reduction and hence needed mixing improvements. Another cluster of customers had exhausted recipe iterations and were simply trying to mix their compounds faster. In fact, numerous customers expressed their desire to move to single stage mixing. As an aside, an internal estimate was performed to determine what single stage mixing meant to a typical sponge producer in terms of time. Table I describes the manufacturing steps necessary to mix a sponge recipe in a two stage and single stage procedure. It was estimated that one pass mixing could reduce mixing time by up to 50%! In addition to this, added benefits of lower manpower to transport masterbatch, no storage of masterbatch and fewer contamination issues all were expected to provide time and cost savings.

Table I

Comparison of Steps Required – Single Stage vs. Two Step Mixing

Procedure	Single Stage	Two Step
Weighing of ingredients	X	X
Transport to mixer	X	XX*
Internal mixing procedure	X	XX**
Dumping on mill	X	XX
Mill/ Stock-blender mixing	X	XX
Straining via Gear pump	X	X
Cooling/ batch-off	X	XX
Slab/ strip storage***	X	X(X)
Typical cycle time (min)	**6 - 9**	**12 - 16**

* Rubber slabs/strips without curing / blowing agents are transported back for second step mixing
** 2 steps: curing / blowing agents are added to mixer during second step
*** With second step mixing, slabs / strips are added directly after cooling or after 24 hrs storage
(extra disadvantages: contamination and logistical issues)

In regard to polymers used, customers utilized three basic ranges of sponge polymers.

1. Medium Mooney (medium Mw) ML(1+4)@125°C = 55-65
2. Clear (non oil extended) high Mooney (high Mw) ML(1+4)@125°C = 75-90
3. Oil extended high Mooney (ultra high Mw) ML(1+4)@150°C = 45-60

Within each of the Mooney ranges, customers experienced different issues or roadblocks when trying to improve and were looking for help. Table II below describes some of the goals and issues.

Table II

Sponge Improvement Actions and Issues acc. Survey

EPDM Category	**Goal**	**Improvement Action**	**Roadblocks**
Medium Mooney	Reduce cost.	Increase filler and plasticizer loading and use higher Mw polymer to compensate for properties.	Poor dispersion.
	Improve properties.	Use higher Mw polymer.	Poor dispersion.
Clear high Mooney	Reduce cost.	Reduce cycle time of 2 pass mix or even go to single pass mix with existing polymer.	Poor dispersion.
	Reduce cost.	Reduce cycle time of 2 pass mix or even go to single pass mix with lower Mw polymer.	Inadequate physical properties.
	Improve aesthetics and reduce defect scrap.	Use lower Mooney polymer.	Inadequate physical properties.
	Improve aesthetics and physical properties.	Use oil extended high Mooney polymer.	Poor dispersion.
	Reduce surface defect scrap.	Increase mixing shear.	Inadequate mixer capability.
Oil extended ultra high Mooney	Improve aesthetics and reduce defect scrap.	Increase mixing shear.	Inadequate mixer capability.

It was clear that goals of improved mixing of sponge compounds, higher quality surface finishes, and more economic compounding dominated the improvement plans of sealing manufacturers. Users of medium Mw polymers were still attempting to change to higher Mw polymers for reasons of cost and performance; similar to trends started over a decade ago, but were limited by the capability of their mixing equipment. The results of their efforts were always poor compound dispersion and higher scrap levels. Users of clear high Mw sponge grades were less concerned about performance gains but were highly focused on mixing improvements and cycle time reduction. Other manufacturers were interested in scrap reduction and reduced surface roughness of their sponge profiles for better aesthetics and quality improvements including sponge producers using oil extended, ultra high Mw grades.

The question was how could a new polymer be designed so that many customers could see a benefit? Could improvements in cost, performance and quality all be addressed simultaneously? Using both these questions and the survey data as guidance, characteristics for a new grade were methodically selected. Since the majority of customers used polymers in the first two Mooney categories, it was decided to focus the new grade design to offer improvements in these areas. With years of sponge polymer development knowledge and several established grades already in the sealing market, certain characteristics were quickly selected while others required greater consideration. The thought process was as follows.

From a customer standpoint, minimizing cost is paramount. This is true for both materials and process. For this reason, sponge seals are typically extruded at very high rates ranging from 20-30 m/min. In order to accommodate this need, sponge compounds must flow well through the extruder and die; must have sufficient strength to support its shape directly after exiting the die; and then must cure extremely fast. This has always dictated a high amount of ENB for ultra fast vulcanization and the incorporation of controlled long chain branching for good collapse resistance and balance of processability and physical properties. Due to stringent OEM demands on long term seal performance and cold temperature requirements, amorphous EPDM polymers are necessary for superior low temperature properties such as compression set. The remaining characteristic of molecular weight, more easily expressed as Mooney viscosity was more difficult to select as it directly affected how customer roadblocks would be addressed. A whole range of viscosities had already been trialed at customers in years past, always with solutions for some but dilemmas for others. Then the question arose, what if the viscosity was modified by simply adding oil to a high Mw polymer. As with all oil extended polymers, the removal of free oil from the mixing chamber reduces the chance of slippage, and increases shear as the compound passes between the rotors, or the rotor and the chamber wall depending on the design of the mixer. The increased shear generated by an oil extended polymer would enable improved dispersion versus clear polymers of similar Mw while the recipe would remain unchanged and therefore have similar physical properties. As described earlier, this concept using ultra high Mw EPDM grades for sponge had already been introduced in the early 2000s. Why not add an extender oil to facilitate improved dispersion of a high Mw polymer, providing the opportunity for more economic single stage mixing combined with high quality surface finish, when compared with polymers of similar or higher molecular weight. Versus polymers of lower molecular weight, it could also provide the opportunity to reduce cost by extending total loading without sacrificing properties or quality. Table III summarizes the polymer characteristics initially proposed and why.

Table III

Initial Polymer Characteristic Proposals

Polymer Characteristic	Properties Affected	Intended Affect On Roadblocks
High Mw	1. Good collapse resistance. 2. Excellent physical properties.	1. Allows higher filler loadings and improved properties vs. medium Mw polymers.
Controlled long chain branching	1. Improved mixing. 2. Good collapse resistance.	
High ENB	1. Ultra fast vulcanization to allow high extrusion rates.	
Amorphous	1. Superior low temperature properties.	
Oil extended	1. Lower apparent viscosity.	1. Easier to mix than clear high Mw grade allowing potential for reduced mixing times or single stage mixing and improved surface finish. 2. As easy to mix as medium Mw grade preventing dispersion problems experienced when developing for higher loadings or improved performance.

With the general structure of the polymer decided, the target values had to be set. While most polymer characteristics were determined based on other established sponge grades and on manufacturing capabilities, the optimum oil level remained an unknown. This prompted the decision for experimentation before a new grade could be commercially produced. It was decided that the experiments should demonstrate the mixing behaviour of the new polymer, and if oil extension could indeed remedy some of the roadblocks experienced by customers, providing evidence that significant improvements such as single stage mixing for example, were possible. Finally it was agreed that the new grade should be compared against a variety of well established sponge polymers preferred by many customers to truly test its abilities. Pilot plant samples were created at levels of 10 phr (sample A) and 15 phr (sample B) oil with the expectation that an improvement in processability would be seen. The commercial polymers included Lanxess produced medium Mw (sample C) and high Mw (sample D) grades as well as a competitor high Mw elastomer (sample E).

EXPERIMENTAL I

Polymer Testing

Polymer attributes for all samples tested are described in Table IV below. All polymers were tested according to ASTM, ISO or internal standards. Those standards are listed accordingly.

Table IV

Polymers Properties – First Trial

Properties	**Units**	**Pilot Sample A**	**Pilot Sample B**	**Commercial Grade C**	**Commercial Grade D**	**Competitor Grade E**	**Test Method**
Mooney Viscosity ML (1+4) 125ºC	MU	68	63	65	est.90	81[1]	ISO 289
150ºC					60		
Ethylene content	wt%	48	48	48	48	58	ASTM D3900
ENB content	wt%	9.0	9.0	9.0	9.0	8.9	ASTM D6047
Molecular Weight Distribution	-	Medium (CLCB)	Medium (CLCB)	Medium	Medium	Bimodal	Internal
Oil content	phr	10	15	-	-	-	Internal

[1] ML(1+8)

Compounding and Physical Testing

Compounds using each of the polymers described above were prepared and mixed in the Harburg Freudenberger laboratory in Germany. The compounds are shown in Table V. The entire recipe can be found in Appendix I.

Table V

Recipes - First Trial

Ingredients	**Compound A**	**Compound B**	**Compound C**	**Compound D**	**Compound E**
Pilot Sample A	110				
Pilot Sample B		115			
Comm.Grade C			100		
Comm.Grade D				100	
Comp. Grade E					100
Carbon Black N550	75	75	75	75	75
Paraffinic Oil	65	60	75	75	75
Calcium Carbonate	40	40	40	40	40
Talc	10	10	10	10	10
Zinc Oxide	7	7	7	7	7
Calcium Oxide	2.5	2.5	2.5	2.5	2.5
Stearic Acid	1	1	1	1	1
Polyethylene glycol	2	2	2	2	2
Process Aid	3	3	3	3	3
Sulphur	1.50	1.50	1.50	1.50	1.50
Accelerators	5.10	5.10	5.10	5.10	5.10
OBSH -75	2.50	2.50	2.50	2.50	2.50
AZTF -75	1.50	1.50	1.50	1.50	1.50
Total PHR	**328.02**	**328.02**	**328.02**	**328.02**	**328.02**

Compounds were single stage mixed in a 5.5 liter, GK 5 Harburg Freudenberger (HF) intermeshing mixer. All experiments used an 80% fill factor and mixing chamber, drop door and rotor temperatures set to 40°C. Ingredient loading into the mixer and ram sweeps was performed manually. Compounds were dumped at 95°C, resulting in an approximate total mixing cycle time of 90 sec. and an effective mixing time of 72 sec. The conditions and mix cycle were recommended by Harburg staff and considered as a good starting point. The mix cycle used is shown in Table VI below.

Table VI

Mix Cycle Procedure – First Trial

Step #	**Step Time (sec.)***	**Total time (sec.)**	**Max Temp (°C)**	**RPM**	**Ram Position**	**Feed Door**	**Drop Door**	**Event**
1	5	5		60	UP	Open	Closed	Addition of Polymer, Filler, Oil
2	5	10		60	UP	Open	Closed	Addition of cure and small ingredients
3	3	13		60	UP	Closed	Closed	Close Feed Door
4	30	43	80 C	60	DOWN**	Closed	Closed	Mix
5	15	58		50	UP	Open	Closed	Ram Clean
6	4	62		50	UP	Closed	Closed	Close Feed Door
7	15	77	95 C	50	DOWN**	Closed	Closed	Mix
8	15	92			Up		Open	Dump

* Step time was approximated based on all mix cycle data
** Ram Force [N/cm^3] = 54

Directly after mixing, compounds were passed three times through a two roll mill at a gap of 5 mm to produce slabs, and were allowed 24 hr relaxation time prior to extrusion. All mixed compounds were extruded on an HF ES 30 Quality testing extruder, then directly fed into a Pixargus Profile Control PRC 20/40 surface roughness analyzer. All compound and physical tests were carried out in the Lanxess laboratory in The Netherlands using ASTM methods.

RESULTS AND DISCUSSION I

Since one of the goals of the experiment was to test if economic improvement could be offered to customers, it was decided to immediately begin mixing all compounds in a single stage manner. Dispersion results for all compounds are shown in Figure 1.

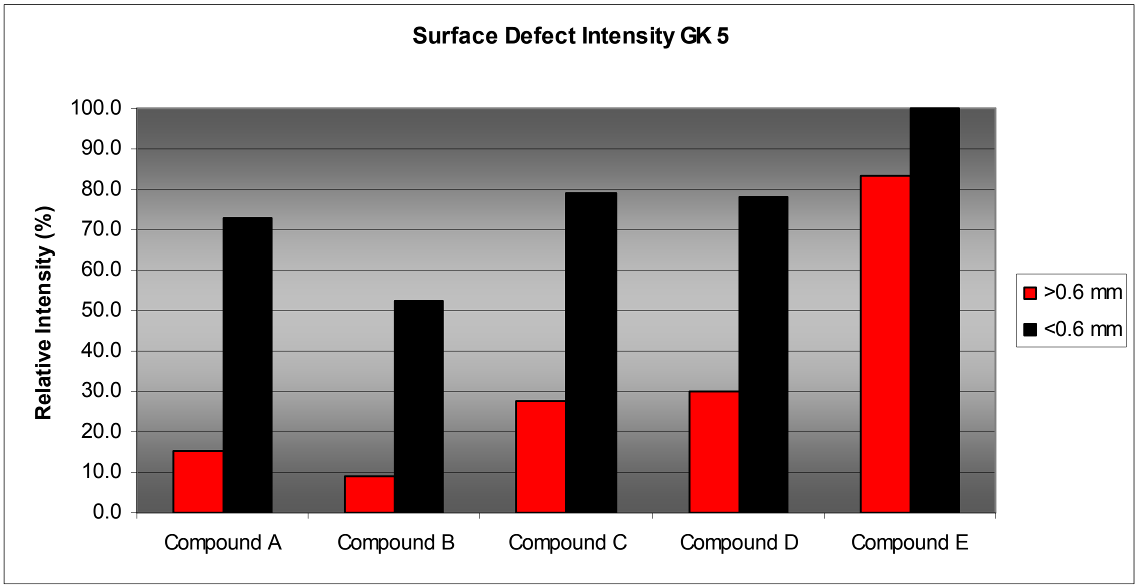


Fig.1. – Surface Defect Comparison of All Compounds Used in First Trial

The Pixargus camera system detects and records surface defects as small as 30 µm on the surface of the extruded ribbon. For a specific length of extruded ribbon it reports the surface area of defects at specific defect diameters as well as the total defective surface area. To simplify the reporting, Figure 1 depicts for each compound, the total defect area measured for defects above and below 0.6 mm in diameter. Areas were normalized and graphed as a relative surface defect intensity. Some simplistic observations were made.

1. The addition of oil to a high Mw polymer helped reduce the defective level in general versus polymers of similar Mw.
2. Compound B with 15 phr oil in the polymer demonstrated the lowest defect level of all compounds.
3. Compound B and Compound D contained polymers with similar Mw yet Compound B exhibited lower defect levels
4. Compound B and Compound C contained polymers with similar apparent viscosity yet Compound B showed lower defect levels.
5. Compound E containing a competitive polymer of similar Mw had the highest defect level.

Observation 3 and 4 were especially important as they demonstrated that Sample B with 15 phr oil had the ability to address two of the roadblocks highlighted by customers during the survey! In a single stage lab mix, oil extended sample B had lowered defect levels versus clear polymers of similar Mw and Mooney. The next question was could performance be also matched or surpassed? Physical tests for all compounds were performed on solid samples and are shown in Table VII.

Table VII

Physical Results All Compounds – First Trial

Test	**Compound A**	**Compound B**	**Compound C**	**Compound D**	**Compound E**
Compound Mooney@100°C					
ML [MU]	46	42.5	33	41	39.9
Mooney Scorch@121°C					
t2 [min.min]	4.43	4.43	4.52	4.65	4.58
t5 [min.min]	4.97	4.97	5.09	5.23	5.14
MDR@180°C					
ML [N.m]	1.2	1.1	0.8	1.1	1.0
MH [N.m]	14.2	13.9	13.5	14.5	13.9
MH-ML [N.m]	13.0	12.8	12.7	13.4	12.9
ts2 [min.min]	0.46	0.45	0.48	0.48	0.47
tc 90 [min.min]	4.04	3.85	4.54	4.19	4.98
Physical Properties*					
Hardness [ShA]	56.8	55.8	56.5	57.4	58.2
Tensile Strength [MPa]	8.9	8.8	7.9	8.8	8.6
M100 [MPa]	1.8	1.8	1.9	2	2.1
M300 [MPa]	5.4	5.2	5.5	6	6.3
Elongation [%]	455	463	415	402	379

*Physical testing performed on solid samples.

The question of performance was easily answered.

1. Compound B with similar Mw polymer to polymer used in Compound D had almost identical physical properties.
2. Compound B using polymer of similar viscosity to Compound C had better tensile and elongation properties.

Preliminary general conclusions were;

1. The addition of oil to a high Mw polymer appeared to generate higher shear in the mixer, thereby improving dispersion and sponge aesthetics, and addressing the earlier mentioned developmental roadblocks
2. The optimum level of oil appeared to be 15 phr (Pilot Sample B). This was based on the fact that Compound B with similar Mw polymer to polymer used in Compound D had almost identical physical properties and Mooney viscosity. An increase in polymer oil level would in all likelihood lower compound viscosity further, causing potential problems with die flow, collapse resistance and profile shape stability. A decrease in oil level would result in poorer dispersion as seen in Figure 1.

This then opened up a realm of possibilities for customers.

1. Customers with state of the art mixing equipment typically used high or ultra high Mw polymers since they had the equipment capability and in some cases single stage mixed. A new oil extended polymer offered the chance for improved dispersion and lower defect levels leading to lower scrap, or faster mixing cycle times at unchanged scrap levels.
2. Customers with less than state of the art or worn equipment typically used medium or high Mw polymers. Users of high Mw polymers were usually forced to mix in two passes to attain required quality levels. A new oil extended polymer could offer users of high Mw polymers lower scrap levels, or the ability to mix faster if not single stage mix. Users of medium Mw polymers in certain cases had the ability to single stage mix. A new oil extended polymer could offer reduced defect levels as seen in Figure 1, but a greater offering would be the possibility of improved properties (as seen in Table VII) or the chance to load the compound more, thereby reducing compound costs.
3. Customers with small technical staff could easily implement a new oil extended polymer, since in many cases, very little formulation change was required and immediate quality or cycle time improvements could be seen.

Based on this, a decision was made to produce a grade on a production scale. The new grade was called Keltan® 6251A and had characteristics as shown in Table VIII.

Table VIII

Polymer Characteristics Keltan® 6251A

Properties	Units	Typical value	Test Method
Mooney Viscosity ML (1+4) 125ºC	MU	63	ISO 289
Ethylene content	wt%	48	ASTM D3900
ENB content	wt%	9.0	ASTM D6047
Molecular Weight Distribution	-	Medium	-
Oil content	wt%	13	Internal
	phr	15	

A second round of trials was then undertaken to further understand the performance of Keltan® 6251A against other polymers. In these experiments, Keltan® 6251A was compared against two competitive grades; one of similar Mw; the other, an oil extended ultra high Mw grade.

EXPERIMENTAL II

Polymer Testing

All polymers were tested in a similar manner as described in the Experimental I section. Polymers used are shown in Table IX.

Table IX

Polymers Properties – Second Trial

Properties	Units	Keltan 6251A	Competitor F	Competitor G	Test Method
Mooney Viscosity ML (1+4) 125ºC	MU	63	73	80	ISO 289
Ethylene content	wt%	48	54	51	ASTM D3900
ENB content	wt%	9.0	10.0	7.7	ASTM D6047
Molecular Weight Distribution	-	Medium (CLCB)	Bimodal	Medium	Internal
Oil content	phr	15	15	-	Internal

Compounding and Physical Testing

Compounds formulations shown in Table X were mixed in a single stage procedure and tested for dispersion using the same methods as described in Experimental section I. Mixing was performed again at the Harburg Freudenberger laboratory, but on the 45 liter mixer to more closely approach a true production environment. In addition, compounds were extruded on production size equipment into profiles at the Lanxess laboratory to gain a better understanding of extrusion processing and part performance behaviour.

Table X

Recipes – Second Trial

Ingredients	Compound B	Compound F	Compound G
Keltan® 6251A	115		
Competitor F		115	
Competitor G			100
Carbon Black N550	75	75	75
Paraffinic Oil	60	60	75
Calcium Carbonate	40	40	40
Talc	10	10	10
Zinc Oxide	7	7	7
Calcium Oxide	2.5	2.5	2.5
Stearic Acid	1	1	1
Polyethylene glycol	2	2	2
Process Aid	3	3	3
Sulphur	1.50	1.50	1.50
Accelerators	5.10	5.10	5.10
OBSH -75	2.50	2.50	2.50
AZTF -75	1.50	1.50	1.50
Total PHR	**328.02**	**328.02**	**328.02**

Compounds were single stage mixed in a 45 liter, Harburg Freudenberger (HF) intermeshing mixer with PES5 rotors. All experiments used an 80% fill factor and mixing chamber, drop door and rotor temperatures set to 31°C. Ingredient loading and mix cycle was run automatically. Compounds were dumped at 100°C, resulting in an approximate total mixing cycle time of 145 sec. and an effective mixing time of 110 sec. Again, the conditions and mix cycle were recommended by Harburg staff and considered as a good starting point. The mix cycle used is shown below in Table XI.

Table XI

Mix Cycle Procedure – Second Trial

Step #	Step Time (sec.)*	Total time (sec.)	Max Temp (°C)	RPM	Ram Position	Feed Door	Drop Door	Event
1	15	15		40	UP	Open	Closed	Addition of Carbon black, Oil
2	20	35		40	UP	Open	Closed	Addition of Polymer, Talc, small ingredients, cure
4	45	80	80 C	40	DOWN**	Closed	Closed	Mix
5	15	95		35	UP	Open	Closed	Ram Clean
6	35	130	100 C	35	DOWN**	Closed	Closed	Mix
7	15	145		35	Up	Closed	Open	Dump

* Step time was approximate based on all data. ** Ram Force [N/cm^3] = 51

Directly after mixing, compounds were passed once through a two roll mill at a gap of 5mm to produce slabs, and were allowed 24 hr relaxation time prior to extrusion. Additional extrusion experiments were executed in the Lanxess laboratory in The Netherlands using a Berstoff 90 mm cold feed extruder. (L/D=16), equipped with a Drato die. A photograph of the die can be seen in Appendix II. The line further consisted of a 6m, 2x 6 kW UHF and a 30 m hot air oven, followed by a 20 m water cooling bath. Line speed was set at 10 m/min and UHF power was controlled such that the UHF exit temperature of the profile was 180 °C.

All compound, physical and finished part tests were carried out in the Lanxess laboratory in The Netherlands using ASTM, ISO or internal methods.

RESULTS AND DISCUSSION II

Defective analysis results from the Pixargus equipment are shown below in Figure 2.

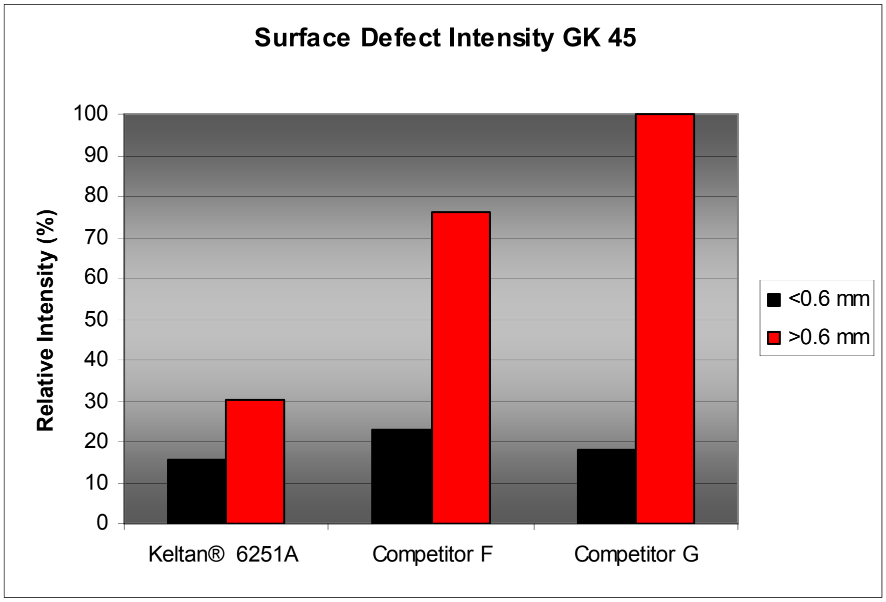


Fig.2. - Surface Defect Comparison of All Compounds Used in Second Trial

Once again, Keltan® 6251A demonstrated the best single stage mixing performance. All polymers seemed to exhibit similar dispersion levels for the smaller defect sizes, but discernible differences were seen at much larger defect sizes. This prompted a closer look at defect intensity at the upper end of the defect size measurements.

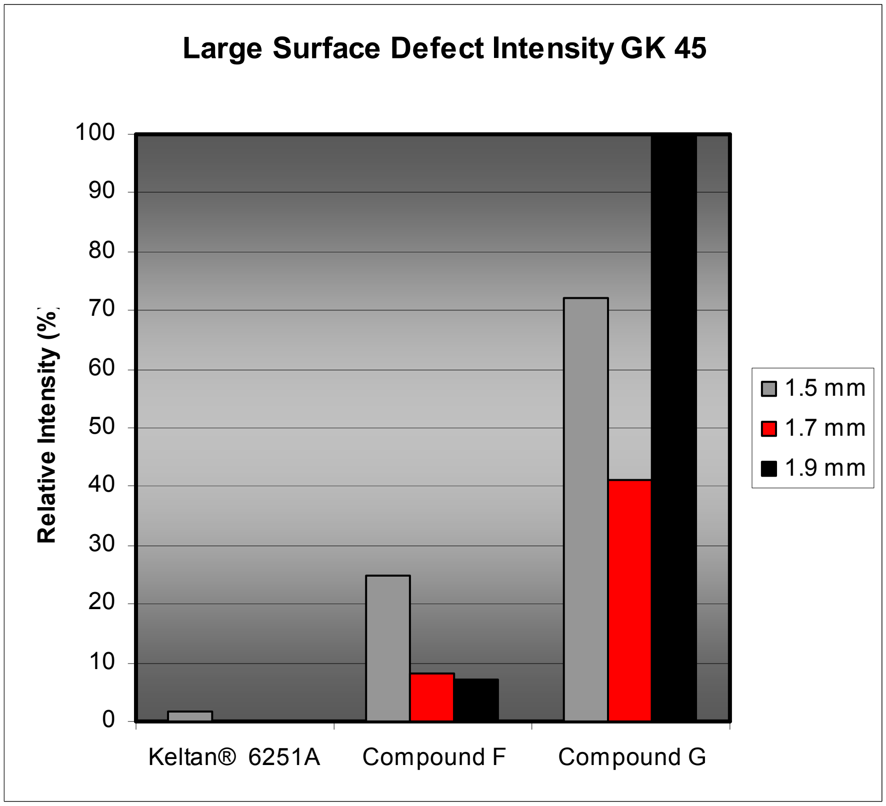


Fig.3. – Large Surface Defect Comparison of All Compounds Used in Second Trial

Figure 3 shows that even at the largest defect sizes measured, Keltan® 6251A demonstrated the best mixing performance. Compound G containing a competitive polymer of similar Mw exhibited very poor dispersion. This was suspected to be linked to its more compact bale structure, as the sole sponge polymer not in friable or semi friable form. Versus Compound F, containing a well-established 15 phr oil extended competitive grade, promoted for its good mixing and surface properties, Keltan® 6251A also excelled. In this case, it had already been suspected that the lower apparent viscosity of Keltan® 6251A might improve mixing performance and this indeed proved true. Photographs of unvulcanized extruded strips were taken, confirming the superior mixing ability of Keltan® 6251A and the Pixargus analysis.

Keltan® 6251A   Compound F   Compound G

Fig.4. - Photographs of unvulcanized extruded strips

However, also expected was that the lower Mw of Keltan® 6251A versus competitor polymer F would provide somewhat lower properties. The compounds produced on the GK 45 mixer in the Harburg Freudenberger lab were then run on the production scale extrusion line in the Lanxess lab in The Netherlands. Profiles were created and tested. Comparison of rheologic and profile physical properties, as seen in Table XII revealed that Keltan® 6251A unexpectedly matched both competitive polymers, with virtually no difference seen. The only difference, although minor, was the improved compression set value from Keltan® 6251A; attributed to its more amorphous nature (due to lower ethylene content) when compared to the competitors grades.

Table XII

Physical Results All Compounds – Second Trial

Test	**Keltan® 6251A**	**Compound F**	**Compound G**
Compound Mooney@100°C			
ML [MU]	41	41	39
Mooney Scorch@121°C			
t2 [min.min]	4.6	4.4	4.7
t5 [min.min]	5.2	5.0	5.3
MDR@180°C			
ML [N.m]	1.1	1.1	1.0
MH [N.m]	14.1	13.7	13.6
MH-ML [N.m]	13.0	12.6	12.6
ts2 [min.min]	0.45	0.43	0.49
tc 90 [min.min]	6.1	6.3	6.9
Profile Physical Properties			
Density (g/cm³)	0.48	0.46	0.47
Tensile Strength [MPa]	1.8	1.7	1.7
M100 [MPa]	0.7	0.7	0.7
Elongation [%]	265	257	273
Compression set (%) 24h@23°C	12	13	15
Compression Load Deflection			
F/10% (N)	8	8	8
F/20% (N)	14	14	15
F/30% (N)	20	20	21
F/40% (N)	44	48	46

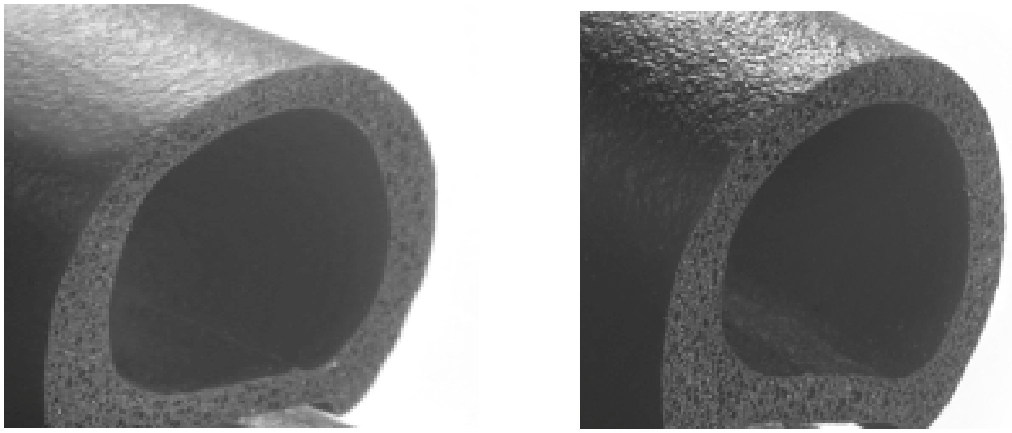
The results from the two experiments comparing Keltan® 6251A against a variety of popular commercial grades provided evidence that the new grade could offer customers the opportunity for improvement in mixing performance, cost and quality. The true test was to now take the grade to the market!

Customer Validation

While the commercialization of Keltan® 6251A is still in its early stages, and little can be divulged from customer trials due to their proprietary nature, the following customer results can be shared.

Customer # 1

This customer replaced their currently used oil extended sponge polymer with Keltan® 6251A. The immediate benefit for them was the improved surface appearance of their profile.



Keltan® 6251A

Oil extended competitive polymer

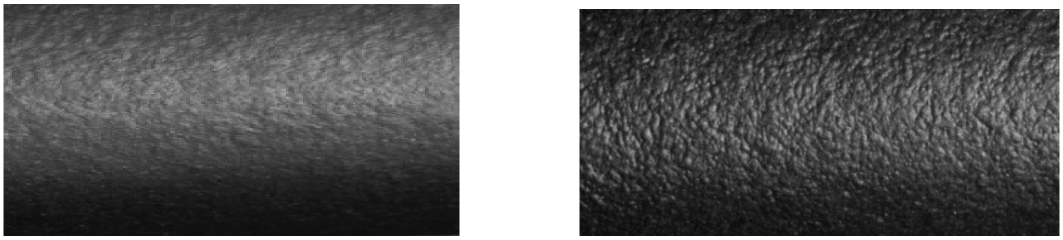


Fig.5. – Photographs of sponge profile surface

Customer # 2

This customer was using a compound based on a blend of an ultra high Mw polymer with a medium Mw polymer. They typically experienced issues with dimensional instability of their extruded profile causing high scrap and associated high production costs. To overcome this problem they would sometimes remix the compound and observe improvement in dimensional control. This indirectly proved their issues were linked to mixing and dispersion. By substituting the blend with Keltan® 6251A they have been able to eliminate the shape instability and reduce their scrap levels without added mixing.

Customer # 3

This customer replaced their existing oil extended polymer with Keltan® 6251A. Compound Mooney viscosity was reduced by 6 points providing better processing; specific gravity was reduced by 10%, generating cost and weight savings; and room temperature compression set at 5 sec. and 60 min. was reduced, bringing the results back into compliance against the specification.

Customer # 4

This customer has verbally communicated that they cannot single stage mix their current high Mw polymer without dispersion problems. However, Keltan® 6251A with similar Mw can be mixed in one pass without dispersion problems, and without the need to cut new extrusion dies or change extruder line settings.

CONCLUSIONS

Lanxess Elastomers recently commercialized a new sponge grade, Keltan® 6251A. Its characteristics were methodically selected based on customer input, technical experience and trials performed in both internal and external laboratories. The proper selection of characteristics positions Keltan® 6251A as a highly versatile and flexible polymer that can offer significant improvement in mixing performance, cost and/or surface quality, when compared against typical sponge grades utilized in the market today. It is considered versatile since it can provide improvements for a variety of customers. In many cases, Keltan® 6251A can facilitate improved dispersion for customers whether they use older mixers or state of the art mixing equipment. The improved dispersion can be translated into reduced mixing cycle times, reduced scrap levels or even single stage mixing ability. In some cases, Keltan® 6251A can assist customers to reduce cost by extending total loading without sacrificing properties or quality. This combination of benefits positions Keltan® 6251A as a leading technical sponge grade that can facilitate the optimization of sponge manufacturing processes, leading to the next generation of high quality automotive sponge seals.

APPENDIX I

Ingredient	Compound A	Compound B	Compound C	Compound D	Compound E
Sample A	110				
Sample B		115			
Commercial C			100		
Commercial D				100	
Competitor E					100
Carbon Black N550	75	75	75	75	75
Flexon 876 Oil	65	60	75	75	75
Calcium Carbonate	40	40	40	40	40
Talc (Mistron R10C)	10	10	10	10	10
Zinc Oxide	7	7	7	7	7
Calcium Oxide	2.5	2.5	2.5	2.5	2.5
Stearic Acid	1	1	1	1	1
Polyethylene glycol	2	2	2	2	2
Struktol WB 16	3	3	3	3	3
Sulphur-80	1.88	1.88	1.88	1.88	1.88
DPG-80	0.50	0.50	0.50	0.50	0.50
MBT-80	1.50	1.50	1.50	1.50	1.50
ZBEC-70	2.14	2.14	2.14	2.14	2.14
Geniplex-70	1.00	1.00	1.00	1.00	1.00
DTDC-80	1.00	1.00	1.00	1.00	1.00
Vulcalent E/C	0.50	0.50	0.50	0.50	0.50
OBSH (Luvomaxx OB-75)	2.50	2.50	2.50	2.50	2.50
AZTF (luvomaxx AZ-75)	1.50	1.50	1.50	1.50	1.50
Total PHR	**328.02**	**328.02**	**328.02**	**328.02**	**328.02**

APPENDIX II

REFERENCES

[1]H.J.H. Beelen, Kautsch. Gummi, Kunst., 52, 406 (1999)

[2] G. Choonoo, G. Vroomen, M. Hatta, T. Odenhamn, Presentation at Automotive Elastomers Conference 2003, EPDM Advancements for Sponge in Automotive Weatherstrips.

[3]M.J. Dees, H.J.H. Beelen and J. Patel, Presentation at the ACS Meeting Rubber Division in Cincinnati, paper # 14 (2000)