Evaluation of Sustainable Bituminous Coal in Elastomer Applications

Erick Sharp – President & CEO, ACE Products & Consulting

Miles Dearth – Technical Project Manager, ACE Products & Consulting

Doug Foster – Director of Technical Services, ACE Products & Consulting

Jaden Slovensky – Lab Zone Coordinator, ACE Products & Consulting

Evaluation of Sustainable Bituminous Coal in Elastomers

Erick Sharp, Miles Dearth, Doug Foster, Jaden Slovensky - ACE *Products & Consulting (Ravenna, Ohio)*

ABSTRACT: Evaluate the use of Sustainable Bituminous Coal, CFI Carbon's Austin Black 325, in elastomer applications. In this evaluation we will explore potential economic, air permeability, compression set, and odor neutralization advantages.

Introduction

Austin Black 325 is a finely divided, below 325 mesh, powder produced from high carbon content, low volatile, sustainable bituminous coal. It has different properties compared to Carbon Black including a lower specific gravity of 1.30 vs. 1.80, a platy ground structure vs. the reinforcing morphology of carbon black (image 1 & 2), and a lower surface area in comparison to carbon blacks. Beyond its carbon composition it is more similar in structure to platy fillers like clay and talc. The specific gravity comparison to other platy minerals, 1.30 vs. 2.50, provides economical and efficiency gains.

Austin Black ground coal was first used in the 1800s by the Austin Powder Co. an early explosives manufacturer founded in Akron Ohio during 1833. AB 325 began to be used as an inexpensive filler in rubber as the automotive industry emerged near the turn of the 20th century. Its use is cataloged in Crude Rubber and Compounding Ingredients, Henry Pearson (1899). The trademark AUSTIN BLACK was first registered in 1962 by the Slab Fork Coal Company and by its successor company Coal Fillers Inc. in 2006. As thermal coal use in power generation declines, it remains one of the cheapest sources of carbon, and the U.S. has the world's largest and cheapest reserves. Sustainable bituminous coal is being utilized for advanced materials in several sectors, including metals, cement, asphalt, roof tiles, lithium-ion batteries, chemicals, and life sciences.

CFI Carbon Products, a Coal Fillers Inc. company, is privately held and headquartered in Bluefield, VA with a plant location also in Tams, WV (Image 3 & 4). CFI Carbon Product states they do not generate any CO2 carbon emissions in their production processing and that all feedstocks are regionally sourced.

Image 1 – Scanning Electron Microscope image of AB 325

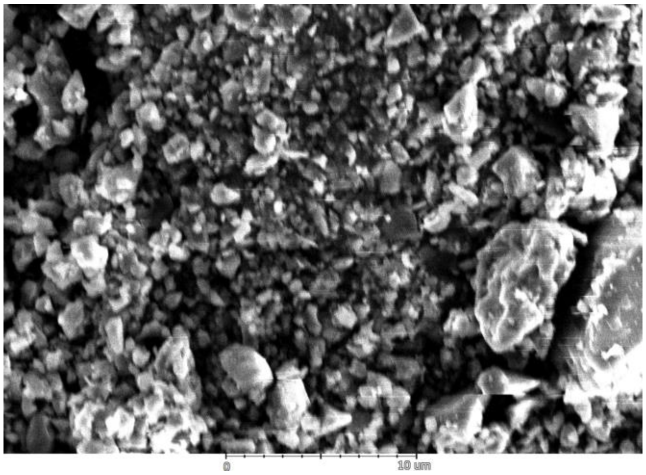


Image 2 – Scanning Electron Microscope image of AB 325

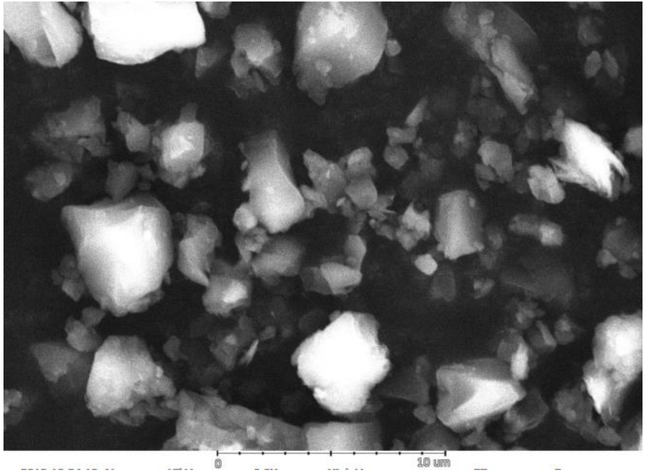


Image 3 – CFI Carbon Operation

Image 4 – CFI Carbon Operations

Pound Volumes Cost Savings vs. Clay and GCC

AB 325 has a specific gravity of 1.30 while soft clay averages a gravity of 2.5 and ground calcium carbonate averages a specific gravity of 2.7. This reduction in weight per volume allows for less material usage when making the same number of products.

An EPDM control compound was selected to evaluate the pound/volume cost savings potentials from using the AB 325 sustainable bituminous coal. In this analysis, the AB 325 was compared to an industry-standard fatty acid-coated soft clay and a 325-mesh ground calcium carbonate (Table 1).

These compounds were evaluated for dispersion (Table 2). The AB 325 achieved similar dispersion values to the fatty acid-coated soft clay. Both the AB 325 and the clay achieved improved results over the 325-mesh ground calcium carbonate (Table 2).

Costing analysis was done based on equal raw material prices for all variants. Dry costs for all three formulations were the same however the gravity of the filler significantly improved the costing of the AB 325 variant (Table 3).

One pound of material from each batch was then used to mold as many compression set buttons as it could yield. This is to simulate the product output achieved with a lower specific gravity compound. The soft clay and ground calcium carbonate batches achieved 66 molded buttons while the AB 325 compound achieved 74 molded buttons (see image 5).

Table 1 – Pound Volume Study Formulations

Material	Variant 1 phr	Variant 2 phr	Variant 3 phr
Royalene 512	100.00	100.00	100.00
N550	80.00	80.00	80.00
Sunpar 2280	70.00	70.00	70.00
Coated Soft Clay	90.00		
325 GCC		90.00	
AB325			90.00
ZnO	5.00	5.00	5.00
Stearic Acid	1.00	1.00	1.00
Sulfur	1.00	1.00	1.00
MBTS	0.50	0.50	0.50
ZDBC 80	1.00	1.00	1.00

Table 2 – Dispersion Analysis

ASTM D773 Dispersion Analysis	**Coated Soft Clay**	**GCC**	**AB 325**
Dispersion Z%	96.5	82.4	93.7

Table 3 – Cost-Saving Calculation

	Clay	**GCC**	**AB 325**
LB / Volume Cost	$2.52	$2.60	$2.03

Image 5 – Comparison of the number of buttons yielded

66 buttons achieved with soft clay and GCC compounds

74 buttons achieved with AB 325 compound

Evaluation in HCR Silicone Compounds

The sensitivity of the cure systems for silicone elastomers limits the types of fillers that can be used. Depending on the residual elements in the filers, they can poison or accelerate the cure system. AB 325 was evaluated in multiple HCR silicone elastomer cure systems as part of this evaluation. Rheology, physical properties, and heat age resistance was then comparatively evaluated.

Because of the slight difference in reinforcement and the economic allowances provided by AB 325's specific gravity, it was substituted into these 50-part filler compounds at 30 parts. This evaluation was performed on DBPH, Dicumyl, Vulcup, DCBP, and platinum cure systems. The AB 325 did cause cure inhibition in the DCBP and platinum systems. DCBP and platinum were removed from further analysis after the rheology screening.

Utilizing the AB 325 in the DBPH screening formulation (Table 4) yielded some benefits. The rheology was not inhibited with the vinyl-specific DBPH peroxide (Table 5). Physical property comparisons to the ground quartz batches were within standard deviation (Table 6). There was a noticeable improvement in heat age tensile loss with the AB 325 (Table 7) while compression set (Table 8) remained sTable. Rheology was rerun after two weeks (Table 9) to determine if the AB 325 accelerated cure decomposition which would result in shelf-life issues. The cure remained sTable and comparatively within the standard deviation, even after two weeks.

The AB 325 yielded similar compatibility with the vinyl-specific Dicumyl peroxide (Tables 10 – 15). Rheology and physical properties remained within standard deviation. An improvement on heat age tensile properties was not discovered with the Dicumyl evaluation. Cure properties did remain sTable across the two-week shelf-life screening.

In the Vulcup evaluation (Tables 16 -21), slight cure inhibition was detected. Both the Ts2 and Tc90 times were extended on the AB 325 batches. Physical properties and heat age properties remained within standard deviation. There was a noticeable improvement in the compression set properties when utilizing AB 325. The two-week rheology results showed the same differential in scorch and cure time that was shown in the original rheology properties. This spread did not increase over time. This means while there would be some slight process changes due to the initial change in cure rate, the overall shelf life of the compound would not be reduced.

Peroxides are notorious for their potent smell, even after the product is fabricated. Depending on the application, this smell can be a deterrent for consumers. The Dicumyl peroxide and DBPH peroxide compounds were comparatively evaluated for odor. The odor of the DBPH after curing was substantially reduced in the compound utilizing the AB 325. Odor absorption is a natural characteristic of activated carbon.

AB 325 was evaluated in a 70-durometer silicone molding compound (Table 22 - 24). This compound utilized a DBPH peroxide cure system. The AB 325 was compared to standard 10-micron ground quartz. It was evaluated both as a 1:1 replacement and as a 0.60 :1 ratio. The objective of this analysis was to determine if lb. /volume cost savings can be obtained while maintaining similar performance properties.

The 1:1 substitution showed an increase in durometer along with a decrease in tensile and elongation properties. The compression set did improve with a 1:1 switch to AB 325. Depending on required specifications, the 1:1 transition could cause compounds to fall out of compliance. With the 0.60 PHR AB 325 substitution per 1 PHR of ground quartz, physical properties equaled out. Durometer and tensile moved within standard deviation while there was a slight improvement in elongation properties. Compression set properties improved substantially.

AB 325 provided substantial savings in the cost analysis for the 70-durometer silicone molding compound. This was a result of the significantly lower specific gravity for AB 325 in comparison to the 10 µm ground quartz. The dry cost per lb. for AB 325 is also lower than that of the ground quartz.

Table 4 – DBPH Evaluation Formulation

Fomulation	10 micron GQ	4 micron GQ	AB 325
RBB-2000-35	100.00	100.00	100.00
10 micron ground quartz	50.00	-	-
4 micon ground quartz	-	50.00	-
AB 325	-	-	30.00
Si-DBPH	1.75	1.75	1.75
Total phr	151.75	151.75	131.75

Table 5 – DBPH Rheology Results

Rheology	10 micron GQ	4 micron GQ	AB 325
MDR (177C for 6 minutes)			
Min (dNm)	0.51	0.47	0.52
Max (dNm)	13.11	12.68	13.21
Ts2	0.32	0.33	0.38
Tc90	0.78	0.69	0.87

Table 6 – DBPH Physical Property Results

Physical Testing	10 micron GQ	4 micron GQ	AB 325
Shore A Durometer	51.50	53.00	51.70
Elongation %	475.43	513.50	490.28
Tensile Strength (Mpa)	5.62	5.80	5.39
Tear Strength Die B (kN/m)	22.87	21.63	22.60
Specific Gravity	1.36	1.36	1.15
Plasticity	158.33	157.67	161.00

Table 7 – DBPH Heat Age Analysis

Heat Age Analysis	10 micron GQ	4 micron GQ	AB 325
% Tensile Loss	-8.63	-9.29	-1.91
% Elongation Loss	-3.93	-4.90	4.00
Duro change	2.20	0.90	2.30

Table 8 – DBPH Compression Set Results

Compression Set	10 micron GQ	4 micron GQ	AB 325
Compression Set 100C for 22 hrs.			
CS Results	2.89	3.08	2.84

Table 9 – Two Week Rheology Rerun

2-week Rheology	10 micron GQ	4 micron GQ	AB 325
Min (dNm)	0.50	0.49	0.51
Max (dNm)	13.38	13.19	14.12
Ts2	0.36	0.37	0.41
Tc90	1.01	0.81	1.01

Table 10 – Dicumyl Evaluation Formulation

Formulation	10 µm GQ	4 µm GQ	AB 325
40 duro silicone base	100.00	100.00	100.00
10 µm ground quartz	50.00	-	-
4 µm ground quartz	-	50.00	-
AB 325	-	-	30.00
Si-DCP	1.75	1.75	1.75
Total phr	151.75	151.75	131.75

Table 11 – Dicumyl Rheology

Rheology	10 µm GQ	4 µm GQ	AB 325
MDR (177C for 6 minutes)			
Min (dNm)	0.55	0.51	0.50
Max (dNm)	13.28	12.91	13.03
Ts2	0.31	0.32	0.38
Tc90	1.26	1.25	1.39

Table 12 – Dicumyl Physical Testing

Physical Testing	10 µm GQ	4 µm GQ	AB 325
Shore A Durometer	51.80	52.60	50.00
Elongation %	406.22	431.50	500.35
Tensile Strength (Mpa)	4.91	5.23	5.16
Tear Strength Die B (kN/m)	21.75	20.52	20.08
Specific Gravity	1.36	1.36	1.15
Plasticity	157.33	151.33	153.33

Table 13 – Dicumyl Heat Age

Heat Age 100C at 70 HRS	10 µm GQ	4 µm GQ	AB 325
Tensile Strength (Mpa)	5.14	5.19	5.32
Elongation %	428.94	475.20	517.44
Shore A Durometer	52.10	54.10	52.60
% Tensile Loss	4.84	-0.64	3.22
% Elongation Loss	5.59	10.13	3.42
Duro change	0.30	1.50	2.60

Table 14 – Dicumyl Compression Set

Compression Set 100C for 22 hrs.	10 µm GQ	4 µm GQ	AB 325
CS Results	2.18	2.23	2.54

Table 15 – Dicumyl 2-week MDR

Repeat MDR after two weeks	10 µm GQ	4 µm GQ	AB 325
Min (dNm)	0.53	0.57	0.54
Max (dNm)	13.27	13.50	13.56
Ts2	0.32	0.31	0.39
Tc90	1.24	1.21	1.34

Table 16 – Vulcup Evaluation Formulation

FORMULARY	10 µm GQ	4 µm GQ	AB 325
40 duro silicone base	100.00	100.00	100.00
10 µm ground quartz	50.00	-	-
4 µm ground quartz	-	50.00	-
AB 325	-	-	30.00
Si-VCP	1.75	1.75	1.75
Total phr	151.75	151.75	131.75

Table 17 – Vulcup Rheology

Rheology			
MDR (177C for 6 minutes)	10 µm GQ	4 µm GQ	AB 325
Min (dNm)	0.51	0.49	0.49
Max (dNm)	14.30	14.02	15.18
Ts2	0.33	0.33	0.41
Tc90	1.64	1.51	2.04

Table 18 – Vulcup Physical Testing

Physical Testing	10 µm GQ	4 µm GQ	AB 325
Shore A Durometer	55.30	55.90	55.40
Elongation %	365.20	399.53	417.95
Tensile Strength (Mpa)	4.93	4.96	5.10
Tear Strength Die B (kN/m)	22.01	21.59	20.96
Specific Gravity	1.36	1.36	1.15
Plasticity	168.67	162.00	168.33

Table 19 – Vulcup Heat Age

Heat Age 100C at 70 HRS	10 µm GQ	4 µm GQ	AB 325
Tensile Strength (Mpa)	5.15	4.91	5.21
Elongation %	364.09	401.89	429.76
Shore A Durometer	55.50	56.60	55.50
% Tensile Loss	4.53	-1.02	2.04
% Elongation Loss	-0.30	0.59	2.83
Duro change	0.20	0.70	0.10

Table 20 – Vulcup Compression Set

Compression Set 100C for 22 hrs.	10 µm GQ	4 µm GQ	AB 325
CS Results	3.94	3.59	2.68

Table 21 – Vulcup 2-week Rheology

Repeat MDR after two weeks	10 µm GQ	4 µm GQ	AB 325
Min (dNm)	0.51	0.50	0.52
Max (dNm)	14.57	14.21	15.45
Ts2	0.33	0.33	0.40
Tc90	1.56	1.49	2.01

Table 22 – Silicone Cost Saving Evaluation Formulation

Material	**1:1 AB 325**	**.60 : 1**	**GQ**
40 Duro Silicone Base	100	100	100
Precipitated Silica	10	10	10
Ground Quartz	0	0	65
AB 325	65	39	0
DBPH-50	1.5	1.5	1.5
Heat Stabilizer	1	1	1
Magnesium Oxide	1	1	1
Pigment	0	0	1
Total PHR	178.5	152.5	179.5

Table 23 – Silicone Cost Saving Physical Testing

Sample ID	**1:1 AB 325**	**.60 : 1**	**GQ**
Durometer	76.3	70.1	71.5
Elongation	219.76	367.76	258.23
Tensile	3.84	5.06	5.31

Table 24 – Silicone Cost Saving Compression Set

Sample ID	**1:1 AB 325**	**.60 : 1**	**GQ**
Compression Set	6.97	5.4	8.07

Table 25 – Silicone Cost Saving Specific Gravity

Sample ID	**1:1 AB 325**	**.60 : 1**	**GQ**
Specific Gravity	1.2333	1.2037	1.4536

Table 26 – Silicone Cost Saving Lb. / Volume Costing

Sample ID	**1:1 AB 325**	**.60 : 1**	**GQ**
LB / Volume Costing	$1.83	$2.08	$2.64

Inner Liner Evaluation

Owing to its platy morphology of stacked graphitic carbon layers, AB 325 as a rubber extender and substitute for carbon black reduces air permeability in a model bromobutyl rubber tire inner liner compound. Extending the formulation by 20 phr and substituting 20 phr for carbon black N660 resulted in reducing air permeability by 50%. As shown previously in the molding examples, the higher volume solids gained by the use of AB 325 also contribute to increased inner liner per pound of rubber compound.

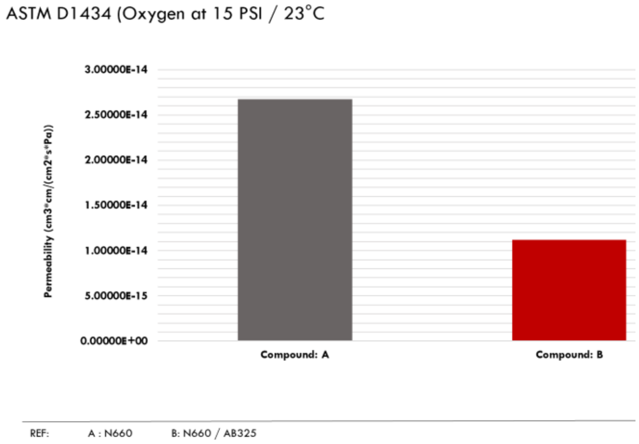
The AB 325 also provides economical advantages in this application. With the specific gravity of the carbon black being 1.80 in comparison to the 1.30 gravity of AB 325 in combination with the cost per pound of AB 325 being half the cost of carbon black, the economical savings are substantial. In the formulation evaluated (Table 27), the AB 325 provided an 18% cost reduction.

Table 27 – Inner Liner Evaluation Formulation

Raw Material	**Control**	**AB 325**
Bromobutyl	100.00	100.00
N660	60.00	40.00
AB 325	0.00	40.00
Homogenizing Agent	7.00	7.00
Aliphatic Resin	4.00	4.00
Magnesium Oxide	0.15	0.15
Napthenic Oil	8.00	8.00
Stearic Acid	2.00	2.00
Zinc Oxide	1.00	1.00
Sulfur	0.50	0.50

MBTS	1.20	1.20
Total PHR	183.85	203.85

Image 6 – Inner Liner Permeability Results



Conclusions

Sustainable bituminous coal provides can provide substantial economic benefits in rubber elastomer applications. The combination of low specific gravity and a low material cost, yields cost savings in most applications.

Despite being a natural carbon-based product, AB 325 is compatible with vinyl-specific peroxide systems. The ability to interact in vinyl-specific peroxide systems allows the AB 325 to be a filler option in silicone applications. In addition to the cost savings advantages, in peroxide applications, the AB 325 provides odor absorption benefits. These benefits can mitigate consumer distaste for unpleasant peroxide odors.

The platy structure of AB 325 helps provide a physical barrier to moisture and gas penetration. This advantage is magnified in applications where AB 325 is used as a partial substitution for carbon black.

Published in Rubber World March 2022