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CARBON BLACK CONTAINING EPDM COMPOSITIONS HAVING EITHER A HIGH GLOSS OR A TEXTURED MATTE FINISH
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- (56) Prior Art Documents
WO 93/10195
WO 93/10194
WO 91/17114

(57) We have discovered EPDM compositions containing certain carbon blacks that yield profiles having either high gloss or textured matte finishes when extruded or shaped. The EPDM compositions comprise ethylene-propylene diene monomer (EPDM) and a specified carbon black, which when incorporated into the EPDM will provide articles having a desired high gloss or textured matte finish.

The amount of carbon black in the EPDM composition is such that the EPDM composition will, when extruded or shaped, yield profiles having a high gloss or textured matte finish. The amounts of carbon black utilized range from 100 to about 300 parts by weight for each 100 parts by weight of EPDM. It is, however, preferred to use amounts varying from about 150 to about 250 parts by weight of carbon black per 100 parts by weight of EPDM.

CLAIM

1. An EPDM composition comprising EPDM and a carbon black having a CTAB of $10 \text{ m}^2/\text{g}$ to $70 \text{ m}^2/\text{g}$ and a ratio of DBP/CTAB less than 1.5, wherein the carbon black is present in an amount of 100 to 300 parts by weight carbon black per 100 parts by weight EPDM.
5. The EPDM composition comprising EPDM and a carbon black having a CTAB of $10 \text{ m}^2/\text{g}$ to $30 \text{ m}^2/\text{g}$ and a ratio of DBP/CTAB greater than about 4, wherein the carbon black is present in an amount of 100 to 300 parts by weight carbon black per 100 parts by weight EPDM.
8. An EPDM composition comprising EPDM and a carbon black having a CTAB of $30 \text{ m}^2/\text{g}$ to $70 \text{ m}^2/\text{g}$ and a DBP greater than about 125 cc/100g, wherein the carbon black is present in an amount of 100 to 300 parts by weight carbon black per 100 parts by weight EPDM.

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(54) Title: CARBON BLACK CONTAINING EPDM COMPOSITIONS HAVING EITHER A HIGH GLOSS OR A TEX- TURED MATTE FINISH			
(57) Abstract EPDM compositions comprising EPDM and specified carbon blacks which, when extruded or shaped, exhibit either a highly gloss or a textured matte finish. The carbon black is present in an amount of 100 to about 300, and more preferably about 150 to about 250 parts by weight per 100 parts by weight EPDM.			

CARBON BLACK CONTAINING EPDM COMPOSITIONS
HAVING EITHER A HIGH GLOSS OR A TEXTURED MATTE FINISH

~~This application is a continuation in part of United States Serial Number 07/936,818, filed August 27, 1992.~~

5 FIELD OF THE INVENTION

The present invention relates to EPDM compositions containing certain carbon blacks which when extruded or shaped have either a high gloss finish or a textured matte finish.

BACKGROUND

10 Carbon blacks have been widely utilized as fillers and reinforcing pigments in the compounding and preparation of EPDM compositions. Carbon blacks are generally characterized on the basis of their properties including, but not limited to, their surface areas, surface chemistry, aggregate sizes,
15 and particle sizes. The properties of carbon blacks are analytically determined by tests known to the art, including iodine adsorption number (I_2 No.) and dibutyl phthalate adsorption (DBP).

Heretofore generally known carbon black containing EPDM
20 compositions yield profiles having a dull and flat surface finish when extruded or shaped. In contrast, a smooth glossy finish has a high degree of reflectance when viewed at any angle, in part due to the smooth nature of the surface. Conversely, a textured matte finish has a low degree of
25 reflectance due in part to the rugged nature of the surface. EPDM compositions with high gloss or textured matte finishes are useful in industrial applications such as automotive weather stripping.

SUMMARY OF THE INVENTION

30 We have discovered EPDM compositions containing certain carbon blacks that yield profiles having either high gloss or textured matte finishes when extruded or shaped. The EPDM compositions comprise ethylene-propylene diene monomer (EPDM)



and a specified carbon black, which when incorporated into the EPDM will provide articles having a desired high gloss or textured matte finish.

5 The amount of carbon black in the EPDM composition is such that the EPDM composition will, when extruded or shaped, yield profiles having a high gloss or textured matte finish. The amounts of carbon black utilized range from 100 to about 300 parts by weight for each 100 parts by weight of EPDM. It is, however, preferred to use amounts varying from about 150
10 to about 250 parts by weight of carbon black per 100 parts by weight of EPDM.

EPDM is well known and is commercially available from a variety of sources. The EPDM composition of the present invention may be produced by techniques generally known to
15 those of ordinary skill in the art for mixing rubbers or polymers and carbon black. As will be understood by those of ordinary skill in the art the high gloss and textured matte finish EPDM compositions of the present invention are produced after extruding or otherwise shaping the carbon black-EPDM
20 mixture.

The high gloss or textured matte finish EPDM compositions of the present invention are advantageous for use in applications where a high gloss or textured matte finish is desirable. One such application is automotive
25 weatherstripping.

Other advantages of the present invention will become apparent from the following more detailed description of the invention.

DETAILED DESCRIPTION OF THE INVENTION

The EPDM compositions of the present invention which will yield a high gloss finish comprise EPDM and a carbon black selected from the group consisting of carbon blacks having a CTAB of about 10 m²/g to about 30 m²/g and a ratio of DBP/CTAB greater than about 4, preferably a CTAB of about 25 m²/g and a ratio of DBP/CTAB of about 5.3; and carbon blacks having a CTAB of about 30 m²/g to about 70 m²/g and a DBP greater than about 125 cc/100g, preferably a DBP of about 125 cc/100g to about 160 cc/100g, and most preferably a CTAB of about 47 m²/g and a DBP of about 126 cc/100g.

The EPDM compositions of the present invention which will yield a textured matte finish comprise EPDM and a carbon black having a CTAB of about 10 m²/g to about 70 m²/g and a ratio of DBP/CTAB less than about 1.5, preferably a CTAB of about 45 m²/g and a ratio of DBP/CTAB of about 1.0, and most preferably a CTAB of about 67 m²/g and a ratio of DBP/CTAB of about 0.7.

The amount of carbon black in the compositions of the present invention is sufficient to allow the EPDM composition to yield a profile having either a gloss or a textured matte finish after extruding or shaping. The amounts of carbon black to be incorporated into the EPDM compositions of the present invention range from 100 to about 300 parts by weight carbon black per 100 parts by weight EPDM, preferably from about 150 to about 250 parts by weight carbon black per 100 parts by weight EPDM.

The high gloss or textured matte finish EPDM compositions of the present invention may also contain conventional additives such as oils, stabilizers, oxides, acids, curing agents etc. known to those of ordinary skill in the art as useful for incorporation into, and during the formation of, EPDM compositions.

The high gloss or textured matte finish EPDM compositions of the present invention may be produced in any manner known to those of ordinary skill in the art for producing EPDM compositions incorporating carbon blacks. One method for producing the high gloss or textured matte finish compositions

of the present invention, utilizing a Banbury BR mixer, is as follows.

The Banbury BR mixer is started and maintained at a temperature of 45° C and a rotor speed of 77 RPM. EPDM is added to the mixer and mixed for approximately 30 seconds. Oil, and preferably zinc oxide and stearic acid, or compounds having similar properties, are added to the EPDM and mixed for approximately 2 additional minutes. The carbon black is added to the mixture and the temperature of the mixing chamber is cooled and maintained at a temperature of below approximately 135° C. The carbon black - EPDM mixture is mixed for approximately 4 1/2 minutes and then curing agents are added to the mixture. The resulting mixture is mixed for approximately 1 1/2 minutes while the temperature is maintained at below approximately 135° C. The compound is then discharged from the mixer and extruded, or otherwise formed and cooled to form the high gloss or textured matte finish EPDM compositions of the present invention. By way of example, the batch compound may be extruded on a Brabender extruder at 50 RPM and 100° C through a die having an opening of 1 inch in width and 0.020 inch in height. This will yield a profile having a high gloss or textured matte finish.

Although one method for producing the high gloss or textured matte finish EPDM compositions of the present invention has been described, the high gloss or textured matte finish EPDM compositions of the present invention may be produced by other methods for producing polymer or rubber compositions known to those of ordinary skill in the art.

The following testing procedures are used in the determination and evaluation of the analytical properties of the carbon blacks utilized in the following examples.

The DBP (dibutyl phthalate adsorption value) of the carbon black was determined according to the procedure set forth in ASTM D2414. The I₂No. (iodine adsorption number) of the carbon blacks was determined according to ASTM D1510. CTAB (cetyl-trimethyl ammonium bromide) absorption value of the carbon blacks was determined according to ASTM Test

Procedure D3765-85. Tinting strength (Tint) of the carbon blacks was determined according to ASTM Test Procedure D3265-85a. The CDBP (dibutyl phthalate adsorption value) of the crushed carbon blacks was determined according to the procedure set forth in ASTM D3493-86.

The effectiveness and advantages of the present invention will be further illustrated by the following example.

EXAMPLE

Eighteen EPDM compositions were prepared incorporating six different carbon blacks at each of three different loading levels into EPDM. Carbon blacks A and B were utilized to produce textured matte finish EPDM compositions of the present invention RA1 - RA3 and RB1 - RB3, respectively. Two control carbon blacks, C and D, were utilized to produce EPDM compositions RC1 - RC3 and RD1 - RD3, respectively. Carbon blacks E and F were utilized to produce high gloss EPDM compositions of the present invention RE1-RE3 and RF1-RF3, respectively. The analytical properties of the carbon blacks A-F were as shown in Table 1 below:

Table 1

Carbon Blacks

	A	B	C	D	E	F
I ₂ No. (mg/g)	40	63	30	35	22	51
CTAB (m ² /g)	45	67	30	36	25	47
DBP (cc/100g)	44	49	69	91	132	126
CDBP cc/100g	43	45	59	72	77	83
Tint (%)	80	101	52	57	33	60
DBP/CTAB	1.0	0.7	2.3	2.5	5.3	2.7

EPDM compositions RA1, RA2, RA3; RB1, RB2, RB3; RC1, RC2, RC3; RD1, RD2, RD3; RE1, RE2, RE3; and RF1, RF2, RF3 were prepared utilizing each of the carbon black samples in amounts of 150 parts by weight, 200 parts by weight and 250 parts by weight

parts by weight, 200 parts by weight and 250 parts by weight in the EPDM Compound Formulation shown below in Table 3. The carbon black and amount by weight of the carbon black utilized in the EPDM compositions were as shown in Table 2.

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Table 2

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EPDM COMPOSITION	CARBON BLACK	AMOUNT BY WEIGHT
RA1	A	150
RA2	A	200
RA3	A	250
RB1	B	150
RB2	B	200
RB3	B	250
RC1	C	150
RC2	C	200
RC3	C	250
RD1	D	150
RD2	D	200
RD3	D	250
RE1	E	150
RE2	E	200
RE3	E	250
RF1	F	150
RF2	F	200
RF3	F	250

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TABLE 3 - EPDM COMPOUND FORMULATION

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INGREDIENT	PART BY WEIGHT
EPDM	100
Carbon Black	150/200/250
Sunpar 2280 Oil	100
Zinc Oxide	5
Stearic Acid	1
TMTDS	2.7
Butyl Zimate	2.7
Methyl Zimate	2.7
Sulfur	0.5
Sulfasan R	1.7

EPDM -EXXON VISTALON® 5600, manufactured and sold by EXXON Corporation, Wilmington, Delaware;

Sunpar 2280 - A trademarked oil manufactured and sold by Sun Oil Company;

TMTDS - tetramethylthiuram disulfide;

Butyl Zimate - a trademarked zinc

40

dibutyldithiocarbamate manufactured and sold by
R. T. Vanderbilt Co.;

Methyl Zimate - a trademarked zinc
dimethyldithiocarbamate manufactured and sold by
5 R. T. Vanderbilt Co.;

Sulfasan R - a trademarked 4,4' - dithiodimorpholine,
manufactured and sold by Monsanto, Co., St. Louis,
Missouri.

The EPDM compounds were produced as follows. A Banbury BR
10 mixer was started and maintained at a temperature of 45° C
and a rotor speed of 77 RPM. EPDM was added to the mixer
and mixed for approximately 30 seconds. The Sunpar 2280
oil, zinc oxide and stearic acid, were added to the EPDM
and mixed for approximately 2 additional minutes. The
15 carbon black was added to the mixture and the temperature
of the mixing chamber was cooled and maintained at a
temperature of below approximately 135° C. The carbon
black - EPDM mixture was mixed for approximately 4 1/2
minutes and then the TMTDS, Butyl Zimate, Methyl Zimate,
20 Sulfur and Sulfasan R products were added to the mixture.
The resulting mixture was mixed for approximately 1 1/2
minutes while the temperature was maintained at below
approximately 135° C. The batch compound was then
discharged from the mixer and extruded on a Brabender
25 extruder at 50 RPM and 100°C. through a die having an
opening of 1 inch in width and 0.020 inch in height to
produce the EPDM compositions RA1-RF3 in the form of
profiles.

Upon visual inspection, the finish of each of the EPDM profiles was as follows:

EPDM COMPOSITION	FINISH
RA1 RA2 RA3	Textured Matte Textured Matte Textured Matte
RB1 RB2 RB3	Textured Matte Textured Matte Textured Matte
RC1 RC2 RC3	Dull and Flat Dull and Flat Dull and Flat
RD1 RD2 RD3	Dull and Flat Dull and Flat Dull and Flat
RE1 RE2 RE3	High Gloss High Gloss High Gloss
RF1 RF2 RF3	High Gloss High Gloss High Gloss

These results show that the EPDM compositions RA1-RA3 and RB1-RB3 of the present invention have a textured matte finish whereas EPDM compositions RE1-RE3 and RF1-RF3 have a high gloss finish. Samples produced with the control carbon blacks, RC1-RC3 and RF1-RF3, have a dull and flat surface appearance which is neither highly glossy nor textured matte.

It should be clearly understood that the forms of the present invention herein described are illustrative only and are not intended to limit the scope of the invention.

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. An EPDM composition comprising EPDM and a carbon black having a CTAB of $10 \text{ m}^2/\text{g}$ to $70 \text{ m}^2/\text{g}$ and a ratio of DBP/CTAB less than 1.5, wherein the carbon black is present in an amount of 100 to 300 parts by weight carbon black per 100 parts by weight EPDM.
2. The EPDM composition of claim 1 wherein the carbon black is present in an amount of 150 to 250 parts by weight carbon black per 100 parts by weight EPDM.
3. The EPDM composition of claim 1 wherein the carbon black has a CTAB of about $45 \text{ m}^2/\text{g}$ and a ratio of DBP/CTAB of about 1.0.
4. The EPDM composition of claim 1 wherein the carbon black has a CTAB of about $67 \text{ m}^2/\text{g}$ and a ratio of DBP/CTAB of about 0.7.
5. The EPDM composition comprising EPDM and a carbon black having a CTAB of $10 \text{ m}^2/\text{g}$ to $30 \text{ m}^2/\text{g}$ and a ratio of DBP/CTAB greater than about 4, wherein the carbon black is present in an amount of 100 to 300 parts by weight carbon black per 100 parts by weight EPDM.
6. The EPDM composition of claim 5 wherein the carbon black is present in an amount of 150 to 250 parts by weight carbon black per 100 parts by weight EPDM.
7. The EPDM composition of claim 5 wherein the carbon black has a CTAB of about $25 \text{ m}^2/\text{g}$ and a ratio of DBP/CTAB of about 5.3.
8. An EPDM composition comprising EPDM and a carbon black having a CTAB of $30 \text{ m}^2/\text{g}$ to $70 \text{ m}^2/\text{g}$ and a DBP greater than about $125 \text{ cc}/100\text{g}$, wherein the carbon black is present in an amount of 100 to 300 parts by weight carbon black per 100 parts by weight EPDM.



9. The EPDM composition of claim 8 wherein the carbon black is present in an amount of 150 to 250 parts by weight carbon black per 100 parts by weight EPDM.
10. The EPDM composition of claim 8 wherein the carbon black has a DBP of 125 cc/100g to 160 cc/100g.
11. The EPDM composition of claim 8 wherein the carbon black has a CTAB of about 47 m²/g and a DBP of about 126 cc/100g.

DATED this 29th day of March, 1996.

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CJH/ML DOC 7 AU4839593.WPC

INTERNATIONAL SEARCH REPORT

International Application No
PCT/US 93/08070

A. CLASSIFICATION OF SUBJECT MATTER
IPC 5 C08K3/04 C08L23/16

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 5 C08K C08L C09C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P, X	DATABASE WPI Week 9311, Derwent Publications Ltd., London, GB; AN 93-088771 & JP, A, 05 032 827 (TOYO RUBBER INC. CO., LTD) 9 February 1993 see abstract	1
A	--- CHEMICAL ABSTRACTS, vol. 112, no. 16, 16 April 1990, Columbus, Ohio, US; abstract no. 141037k, see abstract & JP, A, 01 278 342 (TOYODA GOSEI CO., LTD.) 8 November 1989	1, 5, 8
X	--- WO, A, 91 17114 (CABOT CORPORATION) 14 November 1991 see page 8, line 1 - line 16; claims --- -/--	8

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

* Special categories of cited documents :

- *A* document defining the general state of the art which is not considered to be of particular relevance
- *E* earlier document but published on or after the international filing date
- *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- *O* document referring to an oral disclosure, use, exhibition or other means
- *P* document published prior to the international filing date but later than the priority date claimed

- *T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- *X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- *Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- *&* document member of the same patent family

Date of the actual completion of the international search

23 March 1994

Date of mailing of the international search report

- 6. 04. 94

Name and mailing address of the ISA

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Authorized officer

DE LOS ARCOS, E

INTERNATIONAL SEARCH REPORT

I. International Application No

PCT/US 93/08070

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P,X	WO,A,93 10195 (CABOT CORPORATION) 27 May 1993 see claims; example 2 ---	8
P,A	WO,A,93 10194 (CABOT CORPORATION) 27 May 1993 see page 3, line 15 - line 28; claims -----	1,5

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US 93/08070

Box I Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:
2. ☐ Claims Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:
3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1. Claims 1-4
2. Claims 5-11

1. ☒ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.
2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:
4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

on Protest

☐ The additional search fees were accompanied by the applicant's protest.

☒ No protest accompanied the payment of additional search fees.

INTERNATIONAL SEARCH REPORT

Information on patent family members

National Application No

PCT/US 93/08070

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
JP-A-01278342	08-11-89	NONE	
WO-A-9117114	14-11-91	JP-A- 4015268 AU-A- 7873591	20-01-92 27-11-91
WO-A-9310195	27-05-93	US-A- 5236992 AU-A- 3061892	17-08-93 15-06-93
WO-A-9310194	27-05-93	US-A- 5229452 AU-A- 2917692	20-07-93 15-06-93