vulcanized vegetable oils

Vulcanized vegetable oils, as the name implies, are vegetable oils which have been cross-linked or vulcanized. The resultant product is a friable, elastic, thixotropic material (flows or promotes flow under mechanical pressure, but does not flow after pressure is removed) commonly used either as a extender or process aid in elastomeric articles. Rubber substitute, vulcanized oil, oil-rubber, and Akrofax are synonyms for vulcanized vegetable oils. For the purpose of this literature, the term Akrofax will refer to vulcanized vegetable oil.

Akrofax extenders were developed in the early days of the Rubber Industry to replace fluctuating natural rubber supplies. Thus the terms "Rubber Substitute" or "Oil-Rubber." Today, Akrofax is used in many types of natural and synthetic rubber formulations for a variety of reasons. Many compounders have solved processing difficulties and developed unique elastomeric properties with Akrofax and consider this material their "secret weapon." The problem with these additives is that they seem to be misunderstood and/or overlooked. The following technical information will explain the different types, properties, benefits and functions of Akrofax and should prove useful as a reference and usage guide for rubber compounders considering Akrofax as an integral rubber compounding ingredient.

Akrofax is produced by heating and reacting particular unsaturated oils with a cross-linker, catalyst, modifier(s) and stabilizer(s). The typical unsaturated oils used in this exothermic reaction are soya, rapeseed and castor oils. All of these oils are basically unsaturated triglycerides with varying amounts of unsaturation that produce different Akrofax properties, most notably color. Common cross-linkers are sulfur, sulfur monochloride, hydrogen sulfide, peroxides and isocyanates. By varying these cross-linkers with slight adjustments in processing procedures, many different properties can be obtained within the following four classifications of Akrofax: Brown, Light, White, and Sulfurless.

types

The classifications of Akrofax are based on the type of cross-linker utilized to produce these materials. Brown and Light (Brown) grades are produced with sulfur and hydrogen sulfide respectively. White and Sulfurless grades are produced with sulfur monochloride and isocyanate respectively.

Browns:

Brown Akrofax is produced by reacting soya or rapeseed oil with sulfur at about 150°C. The product is then cast in pans, cooled slightly and reacted further in ovens. The resulting cake can be ground through tight mills to produce a homogeneous milled crumb product. Due to a difference in the degree of cross-linking of the surface and



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interior sections, it is advantageous to grind the cake thoroughly to make it homogeneous. Mineral oil is included in certain grades to increase softening. Calcium carbonate is included, usually with additional mineral oil, to lower cost and improve grinding fineness. The following are the Brown Akrofax grades with their corresponding typical properties:

Grade	Acetone Extract, %	Ash, %	Free Sulfur, %	Mineral Oil, %	Comments
Akrofax 173	33 - 41	1 max.	0.1 - 0.5	20 - 22	less scorch, low cost, all elastomers
Akrofax 900C	39 - 47	8 - 12	0.3 - 0.7	28 - 30	lowest cost, SBR and NR
Akrofax K	40 - 48	1 max.	0.1 - 0.5	26 - 28	softest brown, good for low duros
Akrofax A Soft	26 - 34	1 max.	0.1 - 0.5	0	general purpose, all stocks
Akrofax A Akrofax SD	21 - 29 32 - 40	1 max. 1 max.	0.2 - 0.6 0.1 - 0.5	0 9 - 11	stock w/ higher tensile, duros SBR, NR, CR moderate-high loadings
Akrofax BS	19 - 27	1 max.	0.1 - 0.5	0	SBR, NR, CR moderate-high loadings
Akrofax 11 LG	9 - 17	1 max.	0.2 - 0.6	0	light color, small part. sz., best quality

LG = low grit

Light:

Light Akrofax, a.k.a. yellow transparent or golden grades, is made from rapeseed oil reacted with hydrogen sulfide. These grades give the most transparency and are recommended for light colors or translucent compounds. These types are not extended with other oils or fillers and do not retard the cure like White Akrofax. Three Light Akrofax grades are available in milled crumb form:

Grade	Acetone Extract, %	Ash, %	Free Sulfur, %	Mineral Oil, %	Comments
Akrofax B	11 - 19	1 max.	0.3 - 0.7	0	standard, work horse grade
Akrofax BR	6 - 14	1 max.	0.3 - 0.7	0	drier, higher duro stocks vs. B
Akrofax SR	22 - 30	1 max.	0.7 - 1.1	0	lower duro, mooney stocks vs.B

Whites:

White Akrofax is made by reacting rapeseed oil with sulfur monochloride at room temperature. They are used in light colored compounds and retard most cures due to the presence of hydrochloric acid created as a byproduct in the reaction. To offset this characteristic, two recommendations are suggested. First, add a magnesia acid acceptor such as MgO or MgOH to scavenge small amounts of HCl. Second, if necessary, increase or add additional accelerators to adjust to the appropriate cure rate. Two White Akrofax grades are available in milled crumb form:

Grade	Acetone Extract, %	Ash, %	Free Sulfur, %	Mineral Oil, %	Comments
Akrofax 9970	3 - 11	16 - 20	< 0.1	0	standard, work horse grade
Akrofax 57	15 - 23	39 - 43	< 0.1	14 - 16	less cost, softer grade, low duro

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Sulfurless:

Akrofax 758 is a specialty Sulfurless grade of Akrofax produced by reacting castor oil with toluene diisocyanate. This grade was designed for the use in millable urethanes. Many compounders cleverly extended its use to compounds that cannot tolerate sulfur such as fluoroelastomers, peroxide cure compounds and plastics such as PVC. Akrofax 758 contains 28 - 30% mineral oil, virtually no ash and is produced in a milled crumb form. Its degree of cross-linking is measured by n-hexane extract method which ranges from 28 - 36%. N-hexane extract is used, instead of acetone extract, because of the high solubility effect acetone has on the hydroxyl group in the castor oil.

Oil Resistant:

Oil resistant Akrofax is based on castor oil reacted with sulfur. These products are designed to be used in oil resistant compounds and are more compatible with polar polymers such as NBR, NBR/PVC, CR, CSM, etc. The D-LG and FA grades are recommended for rubber parts that will be exposed to solvents. The n-hexane extraction method is used to establish the degree of cross-linking for the same reason mentioned above. Three oil resistant grades are available in milled crumb form and without mineral oil or calcium carbonate modifications.

Grade	Acetone Extract, %	Ash, %	Free Sulfur, %	Comments
Akrofax AS-LG	14 - 22	1 max.	0.9 - 1.3	brown type, standard grade
Akrofax D-LG	12 - 20	1 max.	0.4 - 0.8	better oil resistance, least discoloring
Akrofax FA	12 - 20	1 max.	0.3 - 0.7	best oil resistance, fine particle size
LG = low grit				

LG — The designation LG refers to the "Low Grit" production process. In this process the outer surface or crust of the Akrofax is remove and discarded. Only the inner layers are further processed by refined milling techniques. This insures that no large particles are left in the product that may require applicable shear to fully disperse the material. Lower mooney and/or thin rubber compounds such as rollers, printer blankets, laminates, coatings, etc. will benefit from the Low Grit process.

Cake vs. Crumb — Cake Akrofax is a traditional and widely used form. The cake form is derived from the basic vulcanized vegetable oil process. During vulcanization, the outer surface is firm compared to the softer inside or heart of the cake - similar to a loaf of bread. Cake Akrofax has the same firm surface and soft inside. Because of this, cake forms require greater shear in mixing to achieve proper dispersion.

Milled crumb Akrofax is further processed through grinding and milling, reducing the cake to crumb. Vulcanized vegetable oils in the milled crumb form can tolerate less shear in mixing and still achieve proper dispersion. They disperse more quickly and evenly than the cake products.

properties

Solvent Extract:

This is a measure of the degree of cross-linking by adding a ground sample of Akrofax to boiling solvent under reflux. The resultant extract is a combination of unvulcanized oils, partially sulfurized oils and elemental sulfur. Typically, Acetone Extract is reported. For a few grades using castor oil, N-Hexane Extract is reported due to the high solubility of castor oil in acetone. The higher solvent extract number indicates a lesser degree of vulcanization and a greater degree of softening.

Ash:

Most Akrofax contains only a trace of ash. Ash levels above 1% represent the inorganic filler or extender content in the material. Calcium carbonate and other fillers are added to modify the grinding process and reduce costs.

Free Sulfur:

Refers to the amount of unreacted or lightly bound sulfur in the Akrofax. Many years ago this was a concern because the free sulfur and its complexes could be as high as 5%. This level would tend to accelerate the cure when used at high loadings. But due to many manufacturing improvements, the free sulfur content of Akrofax is very low, 0 - 1.3%. Thus, free sulfur content is rarely a concern.

Specific Gravity:

Usually the specific gravity is near 1.04. If this figure is substantially less than 1.03, mineral oil (or some other oil) has been added to the product. If the specific gravity is more than 1.05, a filler such as calcium carbonate has been added.

benefits and functions

What is unique about Akrofax is its ability to promote a vast array of compounding benefits in all types of elastomers and some polyolefins. A number of these benefits can be achieved within the same compound. Even though these products are often overlooked, some rubber chemists look first to Akrofax to alleviate technical challenges while formulating rubber articles. Akrofax is most commonly employed either as a *process aid* or as a *plasticizer/extender*.

Akrofax is utilized to aid numerous types of processing operations to promote the following attributes:

Mixing

speeds incorporation of fillers improves dispersion reduces sticking on mill, rotors reduces trapped air reduces energy consumption

Preforming/Molding

provides dimensional stability in profile extrusions minimizes distortion in open steam cures lowers mixing temperature requirements improves flow under mechanical pressure increases extrusion speed and reduces die swell reduces trapped air and improves degassing improves definition of extruded profiles improves calendering gauge control reduces mold fouling

In mill and banbury mixing operations, Akrofax is considered a dry weigh-up additive that lubricates and "wet-outs" rubber chemicals. Being dry, these materials allow for easy weigh up and addition to rubber stocks. Add Akrofax early in the mixing cycle unless large loadings are required. If more than 25 phr are being added, some compounders prefer to split the addition: half early in the mix cycle and half near the end of the cycle. This procedure allows the stock to remain stiff in the beginning of the mix to promote adequate shear to disperse the rubber additives.

While processing oils speed incorporation of fillers, improve dispersions and lower mixing temperature requirements, possible problems are associated with these oils. Processing oils reduce viscosity, occasionally promote sticking to mills and rotors, and contribute to air entrapment. Due to its polymeric nature, Akrofax reduces tacky stocks and air entrapment. This is especially important to butyl compounds and low durometer stocks. Its polymeric nature is the key to its aid in molding and preforming operations.

Again, Akrofax is vulcanized vegetable oil. Its molecular weight is much higher than process oils. This attribute increases the compatibility and green strength of Akrofax with elastomers versus processing oils. The increase in uncured strength provides dimensional stability in profile extrusions and minimizes distortion in open steam cures. Extrusion applications is the field where Akrofax is used most. Akrofax increases extrusion speeds, improves definition and reduces die swell of extruded parts. This is very important for complex profiles, weatherstrips, erasers, tubings, hoses, etc. Calendered stocks will notice important benefits with the addition of Akrofax. Calendered products' gauge will become more consistent and easier to control. Less blisters, improved definition and smoothness should be observed. All molding operations should notice less mold fouling after numerous heats. This could extend the "clean life" of the mold and contribute to less down time. Open hot air or steam cures and sponge articles use Akrofax to retain shape and minimize distortion of parts. Pressure Sensitive Adhesives use Akrofax to retain the shape of the tape while still plasticizing the compound under pressure. Also, the Akrofax allows the tape to be peeled off its backing surface while reducing any residual tack.

BENEFITS AND FUNCTIONS: c o n t i n u e d

In addition to processing advantages that Akrofaxes can produce, many physical properties can be improved or created by:

- absorbing a large amount of plasticizer
- reducing plasticizer surface migration
- providing unique velvet surface finish
- reducing abrasion resistance
- reducing bloom, extractability
- improving ozone resistance

One of the first uses of vulcanized vegetable oils was as a rubber substitute to reduce the rubber content of the compound. This was done for two reasons: the unreliable availability of natural rubber and to reduce cost. Vulcanized vegetable oils would extend the rubber compound like process oils, but with modest losses in physical properties. Today, the availability of natural rubbers is not a significant issue, but Akrofax is utilized solely or in combination with process oils to extend the compound (lower costs). Commonly, 2 - 2.5 phr of Akrofax can replace 1 phr of process oil to maintain equivalent hardness. Typical loading range from 5 - 30 phr. If higher loading is utilized, other compounding adjustments may be necessary to obtain equivalent properties.

Akrofaxes are used extensively in low duro compounds. They have the ability to absorb large amounts of plasticizer and anchor these oils into the compound. In other words, Akrofax produces very soft compounds (as low as 5 duro Shore A) in which the rubber article does not bleed, bloom or migrate excess oil. This attribute is very important in the preparation of soft rubber compounds used in the Roller, Flexographic and Molded Goods Industries. Products where aesthetics and feel are an important property will utilize Akrofaxes to create a velvety surface finish. Such products include Footwear, Rolls, Molded Goods, etc. A unique application for these materials is their ability to reduce abrasion resistance. Some applications need the rubber part to abrade easily and quickly to get to a fresh layer of rubber. Erasers are produced with Akrofaxes with this property in mind. Up to 400 phr of White Akrofax are used to produce eraser plugs.

When considering Akrofax and how they affect an elastomer's physical properties, in most cases, they act analogous to a plasticizer. In general, tensile, moduli, hardness, and resilience all will decrease. A few compounds experience an increase in tensile strength with less than 15 phr of Akrofax due to an improved dispersion of the fillers and cure system. Flex life, elongation and low temperature brittleness will improve. An important difference between Akrofax and process oils is that Akrofax will affect the properties to a lesser degree; thus making it easier to obtain the necessary properties. Interesting to note that Akrofax also improves weathering and ozone resistance. Due to its unsaturation (double bonds), Akrofax is preferentially attacked by ozone, sparing the elastomer's double bonds. The ozone protection is marginal, therefore chemical antiozonants and waxes should be used if substantial ozone resistance is necessary. In most cases, Akrofax has little or negligible affect on heat aging properties. Test results have shown that sulfur vulcanized Akrofax has affected ETU-cured polychloroprene's heat and set properties. Akrofax will absorb oils or fuels during fluid resistance testing possibly creating positive swell values. Remember these vulcanized oils are polymeric and will absorb fluids without extracting out of the rubber matrix. Again, typical loading of Akrofaxes are 5 - 30 phr; 10 - 15 phr can be added with very little effect on original physical properties.

BENEFITS AND FUNCTIONS: c o n t i n u e d

Below are various compounds and their resultant physical properties with 0, 15, 30 phr of Akrofaxes:

EPDM:		NBR:	
Buna® EP T 2450	100.00	Krynac [®] 34.E50	100.00
N550 Carbon Black	40.00	Huberpol TM 135	50.00
Paraffinic Oil	10.00	DOP	10.00
RGT-M Zinc Oxide	5.00	RGT-M Zinc Oxide	5.00
Antioxidant DQ	1.00	Stearic Acid	1.00
Perkadox® BC-40C	6.00	Antioxidant DQ	2.00
Akrosorb® 19203 (72% T	'AC) 2.00	Akrochem [®] PEG 3350	1.50
Akrofax® 758	(0-15-30)	Akrosorb® 19249 (72% Si-	69) 1.40
		Akrochem [®] MC-98 Sulfur	1.50
		Accelerator MBTS	1.00
		Accelerator TMTM	0.75
		Akrofax® BR	(0-15-30)
NR:		<u>CR:</u>	
SMR CV (60)	100.00	Baypren [®] 210	100.00
N550 Carbon Black	50.00	N770 Carbon Black	45.00
Plasticizer LN	10.00	Aromatic Oil	10.00
RGT-M Zinc Oxide	5.00	Akro-Mag® Bar Green Lab	el 4.00
Stearic Acid	2.00	Akro-Zinc® Bar 85	5.00
Antioxidant S	1.00	Antioxidant S	2.00
Rubbermakers Sulfur	1.50	Stearic Acid	0.50
Accelerator CBTS	1.25	Akroform [®] ETU-22 PM-75	0.55
Accelerator TMTM	0.30	Accelerator TMTD	0.50
Akrofax® 11LG	(0 - 15 - 30)	Akrofax® A	0 - 15 - 30)

Buna® EP, Krynac®, Baypren® - Bayer Corp., Huberpol™ - JM Huber Corp., Perkadox® - Akzo Nobel

EPDM: Cure - 8 mins./177°C				NBR: Cure - 4 mins./177°C			
Akrofax® 758, phr	0	15	30	Akrofax® BR, phr	0	15	30
Hardness, pts.	59	56	55	Hardness, pts.	76	75	74
Tensile Strength, psi	1019	1021	1194	Tensile Strength, psi	3051	3116	2850
Elongation, %	236	269	313	Elongation, %	550	585	580
100% Modulus, psi	253	226	212	100% Modulus, psi	347	333	327
ML(1+4) 100°C	34.2	33.4	34.0	ML(1+4) 100°C	200	147	145
Die Swell, % ¹	31.1	29.5	28.3	Die Swell, % ¹	20.2	19.4	15.8

'MPT running die swell (300 hz) 'RPA-2000, 100°C, strain 0.5, 2000 cpm values

BENEFITS AND FUNCTIONS: continued

NR: Cure - 3 mins./177°C				CR : Cure - 10 mins./177°C			
Akrofax® 11LG, pbr	0	15	30	Akrofax® A, pbr	0	15	30
Hardness, pts.	54	53	51	Hardness, pts.	60	59	58
Tensile Strength, psi	4010	3696	3316	Tensile Strength, psi	2838	2765	2642
Elongation, %	654	677	681	Elongation, %	480	490	462
100% Modulus, psi	241	212	196	100% Modulus, psi	323	323	328
ML(1+4) 100°C	34.3	27.0	25.7	ML(1+4) 100°C	50.1	50.2	52.5
Die Swell, % ¹	24.3	19.1	18.3	Die Swell, % ¹	26.4	22.9	21.7

¹MPT running die swell (300 hz) ²RPA-2000, 100°C, strain 0.5, 2000 cpm values

From a physical property point of view, all four compounds tested showed only marginal changes occurred with the addition of 15 phr of Akrofax. In the case of the polychloroprene compounds, 30 phr of Akrofax A did not produce significant changes in the compound. Of course, the effect that an Akrofax may produce on a material's physical properties is compound and Akrofax specific. But the compounder should feel safe that 10 - 15 phr of most any Akrofax will not exhibit a significant change in a compound's physical properties.

Although Akrofax decreases most physical properties of a compound at higher loadings (> 15 phr), the ease to process the compound generally increases. The test results show the increase in processability of the rubber stocks as one increases the loading of vulcanized vegetable oil. With addition of Akrofax, the mooney viscosity and die swell of a compound will decrease. This modification helps stocks process more efficiently:

- milling faster and improved...
- extruding faster speeds...
- calendering better gauge control ...

conclusion

From the information above, one can see the vast array of benefits in processing and physical properties that can be achieved with the use of Akrofax. This is why Akrofax is utilized in many industries including Roll, Flexographic, Molded Goods, Footwear, Automotive, Adhesive, etc. However, these products are often overlooked by many compounders. This literature is intended to review and remind rubber compounders of the large number of benefits these products produce from solving difficult processing problems to creating unique compound properties. From process aid to plasticizer/extender, Akrofax should be a part of any rubber compounder's arsenal of rubber chemicals. Further details and information are available from Akrochem. Please contact your Technical Salesperson or the Technical Department at Akrochem.



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