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STABILIZATION OF HIGH-PRESSURE POLYETHYLENE BY NEW ANTIOXIDANTS-PRODUCTS OF THE CYCLOALKLATION OF PHENOL AND o-, m-, AND p-CRESOLS AND 1,2-DIHYDRONAPHTHALENE

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Foreign Technology Division
Wright-Patterson Air Force Base, Ohio

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The product of cycloalkylatin of phenol, o cresol, m cresol, and p cresol by 1,2 dihydronaphthalene were recommended as antioxidants for stabilizing high pressure poly ethylene.
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В в	*В в*	V, v	Т т	*Т т*	T, t
Г г	*Г г*	G, g	У у	*У у*	U, u
Д д	*Д д*	D, d	Ф ф	*Ф ф*	F, f
Е е	*Е е*	Ye, ye; E, e*	Х х	*Х х*	Kh, kh
Ж ж	*Ж ж*	Zh, zh	Ц ц	*Ц ц*	Ts, ts
З з	*З з*	Z, z	Ч ч	*Ч ч*	Ch, ch
И и	*И и*	I, i	Ш ш	*Ш ш*	Sh, sh
Й й	*Й й*	Y, y	Щ щ	*Щ щ*	Shch, shch
К к	*К к*	K, k	Ъ ъ	*Ъ ъ*	"
Л л	*Л л*	L, l	Ы ы	*Ы ы*	Y, y
М м	*М м*	M, m	Ь ь	*Ь ь*	'
Н н	*Н н*	N, n	Э э	*Э э*	E, e
О о	*О о*	O, o	Ю ю	*Ю ю*	Yu, yu
П п	*П п*	P, p	Я я	*Я я*	Ya, ya

* ye initially, after vowels, and after ъ, ь; e elsewhere. When written as ё in Russian, transliterate as yё or ë. The use of diacritical marks is preferred, but such marks may be omitted when expediency dictates.

STABILIZATION OF HIGH-PRESSURE POLYETHYLENE BY NEW ANTIOXIDANTS - PRODUCTS OF THE CYCLOALKYLATION OF PHENOL AND o-, m-, AND p-CRESOLS AND 1,2-DIHYDRONAPHTHALENE

S. F. Naumova, M. V. Balykina, and Ye. N. Artsimenya

(Presented by Academician B. V. Yerofeyev, AN BSSR).

Recently particular attention has been gained by the problem of stabilization of polyethylene during the aging process. For the stabilization of dark parts made from polyethylene compounds from the class of amines are recommended in the literature [1-5], and also a mixture of sulfurous compounds with carbon black. For the stabilization of light parts it is recommended to use dicresyl propane [6] and a number of compounds of the type 2,2'-alkylene-bis-(4,6-dialkylphenol) [7, 8]. In work [9] a study is made of the stabilizing action of alkyl- and arylalkylphenols. It is shown that replacement of the alkyl substituent by a phenylethyl in the ortho-position in phenols increases their stabilizing action during the aging of polyethylene. A proposal has been expressed concerning the influence of the phenols of the intermolecular hydrogen bond of the OH-group with substituents on the stabilizing action.

Physico-mechanical properties of 10802-020 high-pressure polyethylene stabilized by antioxidants - products of cycloalkylation of phenol and o-, m-, and p-cresols by 1,2-dihydronaphthalene in the process of rolling at a temperature of 160°C.

Composition	Concentration of antioxidant	Time of rolling	Indices of quality					
			tensile strength, kgf/cm²;	yield stress, kgf/cm²;	relative elongation, %;	$t_{g\delta} \cdot 10^4$	melting index g/10 min;	amount of gel fraction, %;
Non-stablized polyethylene 10802-020;	0	10 min	120	100	543	1,60	2,0	--
		8 hr	102	96	217	41,9	did not flow	--
Polyethylene 10802-020 + + antioxidant - product of cycloalkylation of phenol by 1,2-dihydronaphthalene with MV 225;	1	10 min	117	97	603	4,0	2,29	0
		8 hr	123	7	616	3,7	1,16	0
Polyethylene 10802-020 + + antioxidant - product of cycloalkylation of phenol by 1,2-dihydronaphthalene with MV 301;	0,5	10 min	122	117	540	—	—	—
		8 hr	125	124	542	—	—	—
	1,0	10 min	130	116	570	7,9	--	0
		12 hr	128	119	549	6,8	--	1,6
Polyethylene 10802-020 + + antioxidant - product of cycloalkylation of o-cresol by 1,2-dihydronaphthalene with MV 217 ± 12;	0,5	10 min	119	108	571	7,9	--	0
		8 hr	122	113	565	6,8	--	1,6
	1,0	10 min	119	108	581	4,7	--	0
		8 hr	134	120	577	3,9	--	0
		12 hr	116	116	494	—	—	0
Polyethylene 10802-020 + + antioxidant - product of cycloalkylation of m-cresol by 1,2-dihydronaphthalene with MV 301;	0,5	10 min	134	119	567	2,8	2,0	0
		8 hr	129	116	530	5,2	1,6	0
Polyethylene 10802-020 + antioxidant - product of cycloalkylation of p-cresol by 1,2-dihydronaphthalene with MV 345 ± 15.	1,0	10 min	125	103	480	4,3	--	0
		8 hr	128	119	576	3,8	--	0

It is interesting to explain the effect of the stabilizing action by aryl-substitutes of phenol and cresols on polyethylene in the case of thermooxidative aging.

In this connection we synthesized new antioxidants - aryl phenol and aryl cresols - by means of the reaction of cycloalkylation of phenol and isomeric cresols by 1,2-dihydronaphthalene.

It is necessary to note that neither the synthesis of antioxidants on the basis of products of cycloalkylation of phenol and cresols by dialin nor the investigation of their stabilizing action have been described in the literature up to now. The effectiveness of the stabilizing action of aryl phenols and aryl cresols on the basis of 1,2-dihydronaphthalene (dialin) was determined by comparing the rate of aging of high-pressure polyethylene in the presence of stabilizers with the rate of aging of non-stabilized polyethylene. Brand 10802-020 polyethylene was subjected to accelerated aging by means of rolling on roll mills at 160°C. The rate of aging of polyethylene samples was evaluated based on the changes in physico-mechanical indices during the process of rolling, including relative elongation during fracture, dielectric loss tangent, etc. The results obtained are summed up in the table, from which it is apparent that all the first products of cycloalkylation of phenol and cresols by 1,2-dihydronaphthalene which were synthesized by us are effective stabilizers. The properties of polyethylene after 8-12 hours of accelerated thermal aging on roll mills remain practically constant and are not changed, while for polyethylene without the antioxidant added in 8 hours the dielectric loss tangent increases by 26 times and relative elongation is reduced by 2.5 times (in respect to the initial sample of polyethylene).

It is necessary to note the preference of products of cycloalkylation of p-, m-, and o-cresols by dialin. These products,

thanks to the presence of a substitute for the $-CH_3$ group in the benzene ring of phenol, combine better with polyethylene.

Light stabilized compositions of polyethylene were obtained on the basis of polyethylene and products of the cycloalkylation of phenol and cresols by 1,2-dihydronaphthalene. An investigation of the IR-spectra of absorption of the products of cycloalkylation of phenol and cresols by dialin showed that they all have ortho-substitution in the benzene ring of phenol in respect to the OH-group. Thus it was proven that usually phenols which are dissolved in carbon tetrachloride have one band of absorption in the range of 3600-3610 cm^{-1} corresponding to valence variation of non-associated OH-group. In the aryl phenols and aryl cresols which we synthesized with a dialin substitute in the ortho-position along with the band of absorption in the range of 3620 cm^{-1} a second band appears in the area of 3540-3580 cm^{-1}. This can be related to the OH-group in the ortho-position, bound by a hydrogen bond with π-electrons of the benzene ring. Apparently the high effectiveness of the stabilizing action of the products of cycloalkylation of phenol and cresols by 1,2-dihydronaphthalene is conditioned by ortho-substitution to the -OH-group of phenol and cresols by dialin and the hydrogen bond between the -OH group and the benzene ring. Such a position was noted earlier in work [9] for alkyl-, alkylaryl-substituted phenols.

Thus for the first time in the literature we synthesized new effective antioxidants - stabilizers on the basis of products from the cycloalkylation of phenol and cresols by 1,2-dihydronaphthalene and investigated their stabilizing action on high-pressure polyethylene.

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