Synthesis and Characterization of a New U.V. Absorber for Low Density Polyethylene

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Abstract- A new photostabiliser, U.V. absorber 2, 4 – bis (4-ethylbenzoyl) resorcinol was prepared and characterized by different techniques such as spectroscopic method, elemental analysis, and moleculer weight determination. The absorber was examined for low-density polyethylene and compared with commercial U.V. absorber (Seesorb-100). The new photostabiliser, U.V. absorber showed higher ability to absorb U.V. light and more efficient for photostabilisation for low-density polyethylene, i.e, the optical properties were improved by using this photostabiliser.

I. Introduction

During the last three decades polymers and plastics, production has rapidly increased, but only 40-60 of them are commercially used ^[1]. These affected by economic and environmental implications. For most commercial polymers, photostabilisers are required in order to protect them from the effect of U.V. irradiation ^[2].

Several types of light stabilisers can be employed^[3], a) u.v. screeners b) u.v. absorbers c) excited state quenchers d) free radical scavengers and e)hydroperoxide decomposers. The u.v. absorbers are among the first stabilisers used for polyolefins and other polymers. They have been used either alone or in combination with other light stabilisers. At first, the main combination with nickel derivatives were used, especially for the stabilization of low density polyeythylene films applications in agriculturel and horticulurel^[4].

The mechanism of this absorber is based essentially, on absorption of the harmful u.v. radiation and then dissipating this radiation in a manner that does not lead to photosensitization, i.e dissipation as heat^[5]. A great deal of efforts have been made in recent years for production of photostabilisers with better compatibility and very high absorption coefficient ^[6]. In this work synthesis and characterization of a new ketone are described which has structure of u.v. absorber type by the presence of intramolecular hydrogen bonded and compared with commercial u.v. absorber (Seesorb-100) i.e 2,4 – dihydroxybenzophenone.

II. Experimental:

1- Material:

The useful chemicals are resorcinol and zinc chloride (Flucka), 4-Ethylbenzoyl chloride and Nitromethan (BDH), low-density polyethylene grade 468 was supplied (state company for petrochemical industries), commercial photostabiliser, u.v absorber (Seesorb-100), 2,4-dihydroxybenzophenone was prepared according to the procedure stated in ref [7].

III. Instrumental

Infrared Spectrophotometer SP 3- 300 Pye- Unicam. U.V. Spectrophotometer UV 260 SHIMADZU. Elemental analysier, EA1108 CARLO ERBA. Haake Rhecord Torgue Rheometer (Haake). Automatic Compression press, PHI (Haake). Mercury lamp 200 (Bausch and Lomb).

IV. Preparation of the new u.v absorber

A new photostabiliser, u.v absorber i.e 2,4-bis (4-ethylbenzoyl) resorcinol was prepared as following: A three necked flask equipped with a condenser, mechanical stirrer and thermometer was charged with (0.01 mole) 4-ethylbenzoyl chloride, (0.005 mole) resorcinol, (50.0 ml) nitromethane and (0.015 mole) fresh fused zinc chloride as the lewis acid [8].

The reaction vessel was stirred vigorously and heat to (60 °C), after 3hrs cooled the flask, then added distilled water filtered the product, dissolved in ethanol, then recrystallization, filtered, dried in vaccum oven at (50 °C) for (24 hr) gave yellowish ppt m.p = 95 °C.

4- Preparation of polyethylene films:

samples of the low-density polyethylene, grade 468 containing the new u.v absorber and commercial light stabiliser (Seesorb – 100) were prepared by Rhecord Torgue Rhrometer then with Automatic Compression press to give a film with 1 mm thickness.

V. Results and discussion:

The structure of the new photostabiliser, u.v absorber was confirmed by different techniques such as infrared spectroscopic (I.R), as shown in Fig. (1). The absorption bands and wave numbers of different functional groups are listed in table (1) [9]. Also by (U.V) spectrum as shown in Fig. (2), shows two different peaks at (326 and 295 nm) respectively, the prime band according to formation six member ring between carbonyl group and the ortho hydroxyl group, i.e. charge transfer, the later band must be a $n-\pi^{\ast}$ transition [10].

On other hand, the chemical formula of the new ketone was confimed by elemental analysis (% Calculated C: 77.00, H: 5.88). (% Found C:76.92 , H:5.79). The molecular weight of the new u.v absorber was determined by freezing point-depression technique using benzene as solvent [11], and was found to be 370 ± 5 compared with calculated value 374. According to the suggested structure of this new ketone, photostabiliser, u.v. absorber might be:

$$O = C \qquad O-H$$

$$H \qquad C \qquad C \qquad C \qquad C - C - R$$

$$H \qquad H \qquad H$$

i.e $R=-C_6H_4_C_2H_5$

The study of absorption efficiency for u.v. light by both the new u.v. absorber and commercial u.v. absorber (Seesorb-100) was examined using Lambert-Beers law, shows the superiority of the new u.v. absorber, over the commercial u.v. absorber (Seesorb-100) [12]. Thus, Fig. (2) shows this comparison, as the absorbance and therefore the extinction coefficient values for the synthetic u.v. absorber are larger than those of the commercial absorber (Seesorb-100), this as shown in table (2). The measurments of the light stability behavior of the low density polyethylene containing (0.1 % by weight) of either 2,4-bis (4-ethylbenzoyl) resorcinol or the commercial absorber, one each time, were done by plotting the carbonyl index versus irradiation time (hr s). Thus, the embrittlement time of the resorcinol derivative (375 hr) was found longer than that of the commercial absorber (290 hr), as shown in Fig. (3). These results reflect the high efficiency of the synthetic u.v. absorber for photostabilisation of the low density polyethylene, relatively to that of the commercial absorber (Seesorb-100). This efficiency might due to the higher ability of the resorcinol derivative to absorb u.v. light and to it high compatibility with low-density polyethylene [13].

The photostabilisation mechanism of the new light stabilizer, u.v. absorber might be similar to that of 2-hydroxy benzophenone compound, which is very well established in literature [14-16].

VI. References:

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Table I Absorption L.R bands for the new synthetic u.v absorber and wave numbers (cm⁻¹)

Functional group	Wave number (cm ⁻¹)
Methyl	2980 asy.str
	2950 sy.str
Carbonyl	1620 str
Phenyl	1580
Methylene	1450 bend.def.
	1375 bend.def.

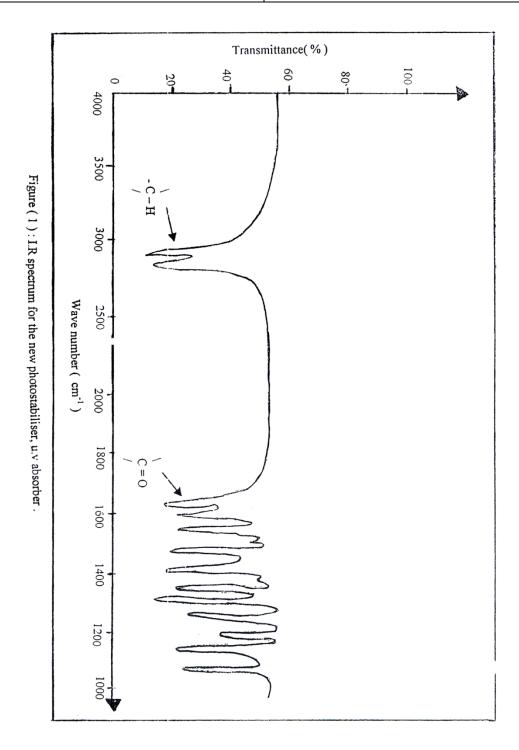
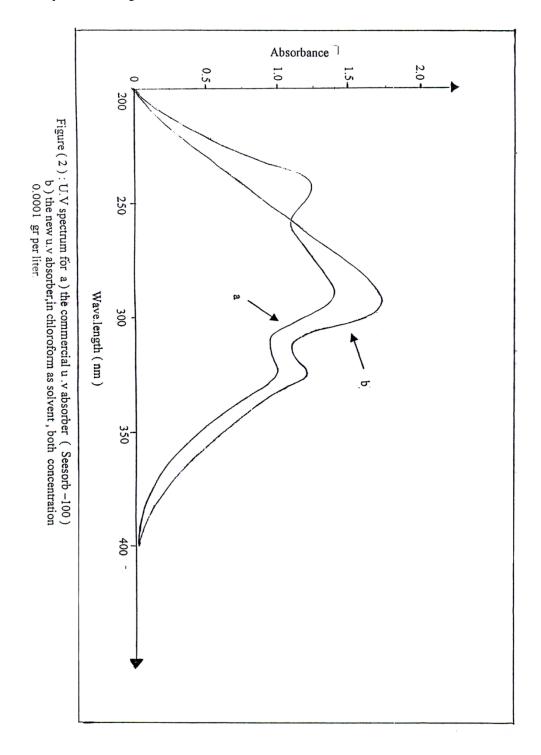


Table III Some characterization for (a) commercial u.v absorber (b) new u.v absorber

Photostabiliser	Wave length max (nm)	Extinction coeff. (cm ⁻¹ g ⁻¹ l)	Transition type
a) Commercial u.v absorber (Seesorb)	325	1.0×10^4	Charge transfer
	289	1.4×10^4	n- π *
	244	1.2×10^4	π -π*
b) New u.v absorber	326	1.2×10^4	Charge transfer
	295	1.7 x10 ⁴	n- π *

Note: Concentration equal to $1 \times 10^{-4} \, g/1$



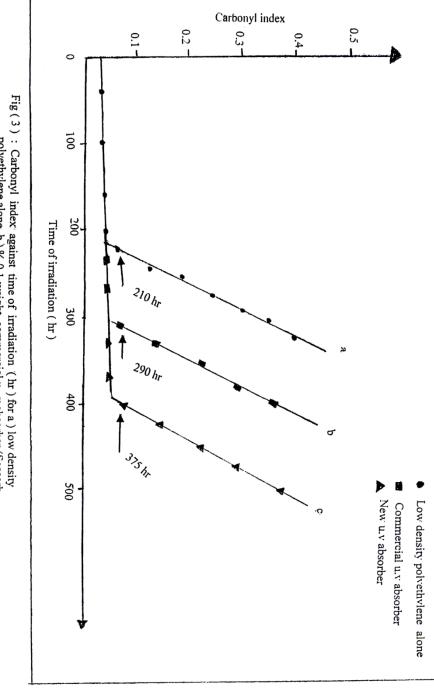


Fig (3): Carbonyl index against time of irradiation (hr) for a) low density polyethylene alone b) % 0.1 weight commercial u.v absorber (Seesorb - 100) with low density polyethylene c) % 0.1 weight new u.v absorber with low density polyethylene.