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Photochemistry and photo-induced co-synergistic polymerisation activities of novel *N*,*N*,-dimethylaminobenzoates and benzamides

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Abstract

Three novel alkylamino ester and seven benzamide derivatives of *N*,*N*-dimethylaminobenzoic acid have been prepared and characterised and their behaviour examined as co-synergists in photocuring of acrylate monomers and prepolymers using 2-propoxythioxanthone as the standard intiator. Absorption, fluorescence and photoreduction analysis have been undertaken in an attempt to relate their photochemistry to their co-synergy in curing. The esters were found to exhibit longer wavelength absorptions than those of the amides and displayed a strong degree of charge-transfer in their lowest excited singlet state. The lowest excited singlet states of all the compounds were found to be essentially $\pi\pi^*$ in character. All the co-synergists provided some degree of photoreduction for the initiator 2-propoxythioxanthone. In general, the amides, apart from compound **3**, were found to be more effective in photoreduction than the esters. The photocuring data for the different co-synergists showed some inconsistencies and appeared to have no relationship with their photochemistry. This is in complete contrast with their primary initiator counterparts where correlations of photochemical and photophysical properties relates well to their photoactivities. The role of the amine co-synergist appeared to be variable and dependent upon structure, electron donation, absorption and perhaps inherent photolytic stability. Overall, the amine esters were found to exhibit enhanced activity compared with the amides and in this way could be related to their greater degree of charge-transfer character enhancing co-synergy to form a more reactive triplet–exciplex with the thioxanthone initiator. © 2000 Elsevier Science B.V. All rights reserved.

Keywords: Photochemistry; Photoinitators; Photopolymerisation; Photocuring

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1. Introduction

The curing of coatings and inks by ultraviolet or visible light has become established technology for many industrial applications, including offset lithographic inks, flexographic inks, screen printing, metal decoration, basecoats for beverages cans, wood coatings, pigmented coatings for textile applications [1–5], coatings exhibiting an stereoscopic pattern [7], pigmented primary and secondary optical fibre coatings [8].

Over the years, several types of photoinitiators have been developed to induce the photopolymerisation or photocrosslinking of acrylated systems. Free radical photoinitiators induce a free radical chain process in which low molecular weight monomers and prepolymers are converted by the absorption of UV–VIS light into highly crosslinked films. Measurements of the rates of curing or crosslinking vary from the simplistic, such as the thumb-screw test, pencil and pendulum hardness methods to the more scientific methodologies involving RTIR spectroscopy and photoDSC [5,9,10]. The basic mechanism for any photocurable free radical system involves the formation of free radical species through the absorption of light by the photoinitiator. The active initiating radical will then add to the monomer, inducing a chain growth polymerisation and eventually termination.

There are two basic categories of photoinitiators, which meet these requirements. The first group involves type I photoinitiators, such as the benzoin ethers, which undergo a direct photofragmentation process (α or less common β cleavage) upon absorption of light and formation of initiating radicals capable of inducing polymerisation. The second group known as type II photoinitiators undergo a primary process of hydrogen atom abstraction from the environment, which may be the resin itself or a solvent, but usually

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a tertiary amine co-synergist is used for enhanced efficiency [1–6]. The reaction usually occurs from the lowest excited state of the ketone, such as benzophenone and depends on the intersystem crossing (ISC) rate, the configuration of the triplet state (n,π^* or π,π^*) and its corresponding energy. Here, the excited triplet-state of the ketone forms an intermediate excited electron transfer complex (exciplex) with the tertiary amine [5,6]. Electron transfer occurs with the subsequent formation of radicals, the amino radical is then believed to be the main initiating radical [5,6].

The UV curing industry commonly uses combinations of types I and II photoinitiators in order to overcome oxygen inhibition and design cost effective photoinitiator blends. Oxygen can quench the lowest excited state of many aromatic ketones. Additionally, the initiating and propagating radicals are also quenched especially on the surface of thin films. The selection of a photoinitiator is of prime importance in the design of UV curing systems, since the polymerisation and/or crosslinking rate depends on the photoinitiator package, and the physical properties of inks and coatings such as flexibility, hardness, scratch, rub and chemical resistance properties are dependent on the degree of cure of the system.

Another importance factor controlling the rate of photopolymerisation of acrylated monomers or prepolymers is the presence of a tertiary amine, which in conjunction with the photoinitiator can be co-synergist [5,6]. Here, the ketone–amine system is believed to form a “triplet–exciplex” through which electron or hydrogen atom abstraction can occur from the amine moiety to form a highly effective alkylamino radical that can induce polymerisation. The structure and nature of the amine is also important in this regard and a number of structural variations have been developed based on the initiator modification. These include *p*-aminobenzophenone derivatives [11–13], 2-alkylaminothioxanthones [14] and acetophenone–amines [15] as examples. Michlers ketone is a well-known specific example, which is strongly synergistic with benzophenone in the photocuring of acrylated systems [16]. In general, the ionisation potential of the amine is important in influencing the ease of electron transfer although steric and other factors also play a role [5,6]. Commercially, the use of solid amine systems is preferable in terms of control over factors such as solubility and diffusion as well as the problem of volatility and smell. To meet this need a solid dimethylaminobenzoate ester was developed (EPD) which has wide applicability in the UV curing industry [5,6]. No work has been undertaken to develop this category of co-synergist. In this regard we report here on the photochemistry and photo-induced polymerisation activities of a range of novel *N*,*N*-dimethylaminobenzoate esters and benzamides using 2-propoxythioxanthone as the primary ketone initiator. In these structures aside from the pendant *p*-dimethylamino group the benzoate esters have been developed to examine the influence of the structure of an additional alkyldimethylamino group on the ester functionality. This compares the effect of an ethyl, 2-propyl and *n*-propyl group. The benzamides on the other hand replace the ester functionality with that of an amide group. The latter contains different primary, secondary and tertiary amine functionalities.

2. Experimental

2.1. Materials

All the *N*,*N*-dimethylaminobenzoate esters and benzamides used in this work are listed in Tables 1 and 2 and were supplied by Great Lakes Ltd., Widnes, UK, as was

Table 1
Structures of *N*,*N*-dimethylaminobenzoate esters

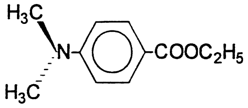
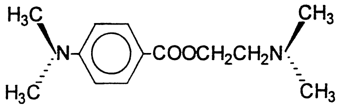
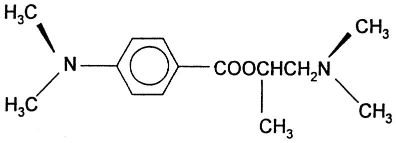
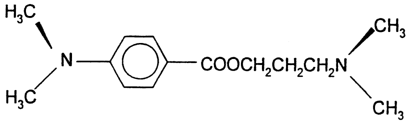
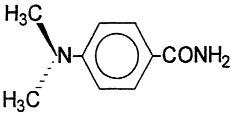
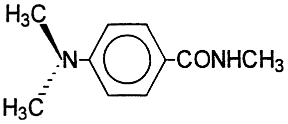
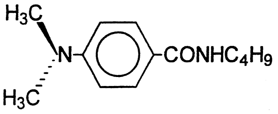
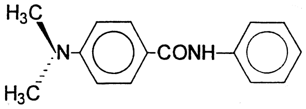
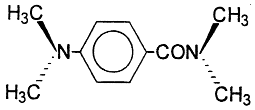
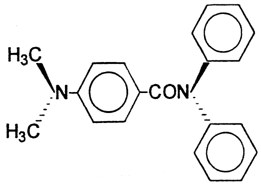
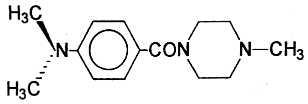
Sample no.	Sample name	Structure
1	Ethyl-*p-dimethylaminobenzoate* (EPD)	
8	Dimehlaminoethyl-*p-dimethylaminobenzoate*	
10	Dimethylamino-2-propyl-*p-dimethylaminobenzoate*	
11	Dimethylamino-3-propyl-*p-dimethylaminobenzoate*	

Table 2
Structures of *N*,*N*-dimethylaminobenzamides

Sample no.	Sample name	Structure
2	*p-Dimethylaminobenzamide*	
3	*N*-methyl-*p-dimethylaminobenzate*	
4	*N*-butyl-*p-dimethylaminobenzamide*	
5	*N*-phenyl-*p-dimethylaminobenzamide*	
6	*N-N*-dimethyl-*p-dimethylaminobenzamide*	
7	*N-N*-diphenyl-*p-dimethylaminobenzamide*	
9	*N*-(4-methylpiperazinyl)-*dimethylaminobenzamide*	

2-propoxythioxanthone. The syntheses of the esters and benzamides were relatively straightforward. The benzoate esters were made by reaction of the corresponding dimethylaminobenzoyl chloride with the respective dimethylaminoalkanolamine while the benzamides were made by reaction of the chloride with the corresponding amine. All the products were purified by recrystallisation from ethanol and flash chromatography on silica with chloroform as the eluant and analysis confirmed through FTIR and FT NMR. Carbon, hydrogen and nitrogen analysis were also confirmed in each case. The melting points of the compounds were all sharp (where applicable) and purity was checked through gas chromatography–mass spectrometry (GC–MS, Table 3). All the compounds and solvents used in this work were purchased from the Aldrich Chemical Company, UK, the latter of spectroscopic grade quality.

2.2. Spectroscopic measurements

Absorption spectra were obtained using a Perkin-Elmer Lambda 16 absorption spectrometer. Fluorescence and phosphorescence excitation and emission spectra were obtained using a Perkin-Elmer LS-50B research luminescence spectrometer. Fluorescence quantum yields were obtained by the relative method using quinine sulphate in 0.05 M sulphuric acid as a standard [17]. The quantum yield of quinine sulphate was assumed to be 0.55. All spectra were corrected using a Perkin-Elmer IBM compatible GEM package with an appropriate file for this purpose.

Table 3
Melting points mass ion peaks of samples

Sample no.	Melting point (°C)	Mass ion peak M^+
1	62–64	193
2	200–201	164
3	143–144	178
4	95–96	220
5	182–184	240
6	92–94	192
7	175–176	316
8	84–85	236
9	65–66	247
10	60–62	250
11	Liquid	250

2.3. Photoreduction quantum yields

Photolysis of the initiator 2-propoxythioxanthone, was determined in anaerobic acetonitrile at 4.2×10^{-4} M in the presence of the compounds **1–11** (Tables 1 and 2) using UV spectroscopy and irradiating with a light wavelength of 391 nm, selected from a Philips high pressure Hg lamp (HB-CS 500 W) and a Kratos GM252 monochromator. Sample cells were thermostatted at 30°C and the solutions were nitrogen ($\ll$5 ppm O_2) saturated. The absorbed light intensity was measured using an International Light Model 700 radiometer previously calibrated by the Aberchrome 540 actinometer [18,19]. Because of product formation during the early stages of irradiation interfering with the quantum yield measurements only subsequent photolysis rates could be measured against light intensity.

2.4. RTFTIR

The initiator, 2-propoxythioxanthone, was dissolved in a minimum quantity (2–3 cm^3 of tetrahydrofuran) followed by mixing with a prepolymer, Actilane 420 (ethoxylated bis-phenol-A-diacrylate) (Akcros Chemicals, Eccles, Manchester, UK) (0.001 M). Traces of solvent were then removed by flushing with argon for 30 min followed by addition of 1% w/w of the co-initiators (Great Lakes, Widnes, UK).

The resin was then placed between pieces of low-density polyethylene using a separator to give a film thickness of 100 μm. The polyethylene holder was then placed on a specular reflectance unit directing the sample beam into the grating system. Two polyethylene film samples were used as the reference. The decrease in absorbance at 1638 cm^{-1} of the vinyl absorption band was then monitored using a Nicolet FTIR spectrometer in real-time mode during irradiation using a fibre optic arrangement (8 mm aperture). The irradiation source used here was a Macam 100 W high pressure Hg lamp. Plots of percentage conversion with time were obtained from which second plots of mol l^{-1} versus time in seconds were obtained. From the initial slopes of the second plots R_p values (mol l^{-1} s^{-1}) were obtained.

2.5. Photocalorimetry

The initial rates of photopolymerisation of lauryl acrylate monomer were determined using a Shimadzu DSC-50 calorimeter using 2-propoxythioxanthone as initator (10^{-2} M) and the amines at 2×10^{-2} M as described previously [20].

2.6. Commercial curing

The photoinitiator, 2-propoxythioxanthone (2.5% w/w) and amines (2.5% w/w) were dissolved in a resin formulation made up of 55% epoxy acrylate oligomer, 20% tripropylene glycol diacrylate and 25% trimethylol propane triacrylate from Akcros Chemicals Ltd., Manchester, UK. Cure speeds of the formulations were assessed using a single 80 W cm^{-1} tube Minicure Unit (Jiga and Lamps Ltd., UK) coated at 50 μm thickness on glass slides and cured to give a pencil hardness of 2H. Cure rates were assessed by pendulum hardness using the Sheenan Pendulum hardness tester (Sheenan Instruments, UK) effective cure.

3. Results and discussion

3.1. Spectroscopic analysis

Aromatic amines possess a strong absorption in the near UV region of the spectrum, which can play a crucial role in controlling the nature of absorption and the free radical processes that exist in parallel with that of the ketone initiator. Table 4 compares the main long wavelength absorption band of the esters and amides as a function of the solvent type. With regard to solvent interactions there are two main effects. The first, relates to the dielectric or polarity of the solvent while the second relates to “anomalous” interactions. The latter involves many solvent types and these have been discussed in detail previously [21]. It is, therefore, often difficult to make predictions regarding solvent shift due to other factors such as hydrogen bonding, solvent conformation and solvent association. From the data shown in Table 4 a number of interesting features are seen. The absorption maxima of the esters are red-shifted from those of the amides as may be expected on the basis of electron donation and conjugation which will be more prevalent in the former case. The only exceptions are the amide derivatives (**5**) and (**7**) which are significantly red-shifted in absorption due to the electron donating effect of the phenyl diphenylamine groups. Generally, the absorption maxima for both sets of ester and amide compounds undergo little shift with solvent polarity and some slight anomalies are evident. For the esters, there is a slight red-shift in the absorption maximum with solvent polarity indicating a strongly mixed $n\pi/\pi\pi^*$ transition with the latter dominating as the lowest state. The extinction coefficients are high (>4.2) indicative of a strong allowed transition and one that is not significantly influenced by solvent polarity. The amide compounds also display a small red shift in absorption maxima with increasing solvent polarity with little change in extinction coefficient. The only anomaly is compound **6** where there is an observable blue shift and this may be due to a TICT (twisted intra-molecular charge-transfer state) preventing delocalisation. Compound **7** is also anomalous in that its absorption maximum is little influence by solvent polarity suggestive of a rigid molecular structure but again the present of a TICT cannot be ruled out.

All the amines and amides studied here were found to be weakly fluorescent and non-phosphorescent. The fluorescence emission maxima and quantum yields are displayed in Table 5 showing again the influence of solvent polarity on

Table 4
UV absorption spectra data

Compound no.	Acetonitrile		*iso*-Propanol		Ethyl acetate		Diethyl ether		Chloroform	
	λ_{max} (nm)	log ε	λ_{max} (nm)	log ε	λ_{max} (nm)	log ε	λ_{max} (nm)	log ε	λ_{max} (nm)	log ε
Esters										
1	307	4.555	309	4.628	305	4.486	311	4.588	302	4.456
8	308	4.512	309	4.570	304	4.493	312	4.453	303	4.346
10	309	4.479	310	4.484	304	4.453	311	4.444	302	4.401
11	309	4.636	310	4.483	306	4.413	311	4.389	302	4.754
Amides										
2	299	4.365	302	4.389	289	4.332	305	4.378	286	4.328
3	290	4.354	298	4.419	285	4.339	300	4.301	283	4.299
4	294	4.540	296	4.419	286	4.412	301	4.378	284	4.330
5	312	4.607	313	4.711	308	4.609	317	4.489	306	4.556
6	280	4.362	284	4.309	297	4.315	287	4.292	278	4.210
7	317	4.405	321	4.378	315	4426	323	4.365	316	4.425
9	282	4.255	288	4.320	281	4.257	289	4 155	279	4.190

the lowest excited singlet state. Generally, the emission maxima of the amines are red shifted from those of the amides a feature associated with the degree of electron mobility and conjugation. Overall, the trend in shift with solvent polarity indicates that the configuration of the lowest excited singlet state is $\pi\pi^*$ in nature. The same is also applicable to the amide compounds but to a lesser degree. However, in the case of compounds **2**, **3** and **4**, there is a strong red-shift in the emission maxima in acetonitrile. All the compounds display a marked blue shift in 2-propanol in their emission maxima from what is predicted by the dielectric function of the solvent $f(D)$. Stabilisation of the excited singlet state by hydrogen bonding may be important here as the ground state absorption maximum is not affected (Table 4). A more quantitative method of comparing solvatochromic shifts is to plot the wavelength or wavenumber as a function of the dielectric strength of the solvent [21]. There have been several attempts at defining the empirical scales of solvent polarity and of these the so-called $E_T(30)$ scale is most applicable [21]. Examples of solvatochromic plots comparing a typical amine with that of an amide compound are shown in Figs. 1 and 2. Both amines **8** and **10** display much greater solvatochromic shifts than the amides **6** and **9**. This suggests that the amines possess a greater degree of charge-transfer in their lowest excited singlet state than the amide compounds and are therefore, more likely to be photosensitive to the polarity of the solvent environment. The amines **8** and **10** are also notable anomalous in terms of solvent shifts in both chloroform and 2-propanol. The quantum yields of fluorescence are relatively low in most of the solvents studies except diethyl ether where solvent perturbations are expected to be minimal.

3.2. Photoreduction quantum yields

The absolute photoreduction quantum yields of the initiator 2-propoxythioxanthone in anaerobic acetonitrile in the presence of the amines and amides are compared in

Table 5
Fluorescence emission spectra data[a]

Compound no.	Acetonitrile		*iso*-Propanol		Ethyl acetate		Diethyl ether		Chloroform	
	λ_{max} (nm)	Φ_F	λ_{max} (nm)	Φ_F	λ_{max} (nm)	Φ_F	λ_{max} (nm)	Φ_F	λ_{max} (nm)	Φ_F
Esters										
1	476.2	0.0231	343.1	0.0081	439.1	0.0671	406.0	0.1741	359.0	0.0114
8	479.5	0.0188	343.7	0.0076	435.5	0.0657	405.9	0.1334	363.5	0.0245
10	487.2	0.0159	344.2	0.0094	444.1	0.0389	403.6	0.1410	364A	0.0208
11	450.3	0.0132	352.8	0.0097	436.8	0.0704	403.9	0.1483	360.7	0.0201
Amides										
2	463.2	0.0306	353.7	0.0115	426.9	0.0934	345.1	0.1461	354.0	0.0031
3	451.68	0.0631	358.2	0.0476	351.3	0.1194	346.3	0.1927	358.5	0.0020
4	447.6	0.0607	366.2	0.0499	351.6	0.1346	345.5	0.1717	360.5	0.0005
5	369.3	0.0075	343.5	0.0019	356.3	0.0051	342.7	0.0064	366~0	0.0023
6	364.3	0.0110	359.5	0.006	348.7	0.0051	340.5	0.0062	358.5	0.0001
7	342.0	0.0037	343.8	0.0033	457.5	0.0060	343.0	0.0110	368.1	0.0085
9	375.8	0.0154	372.2	0.0141	342.1	0.0022	341.1	0.0054	359.7	0.0004

[a] An excitation wavelength of 310 nm was used.

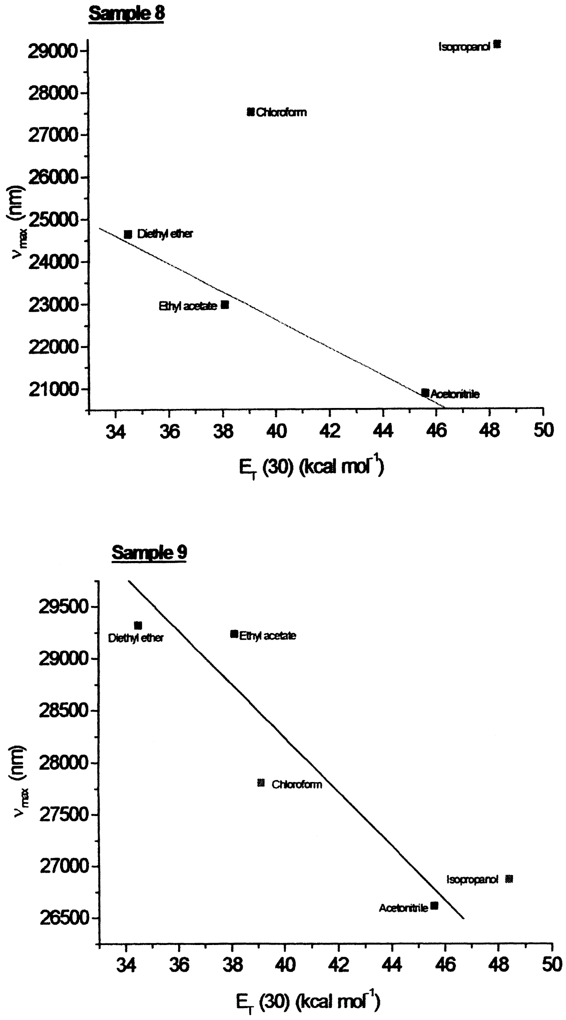


Fig. 1. Solvatochromic plots of wavenumber shift versus $E_T(30)$ for samples **8** and **9**.

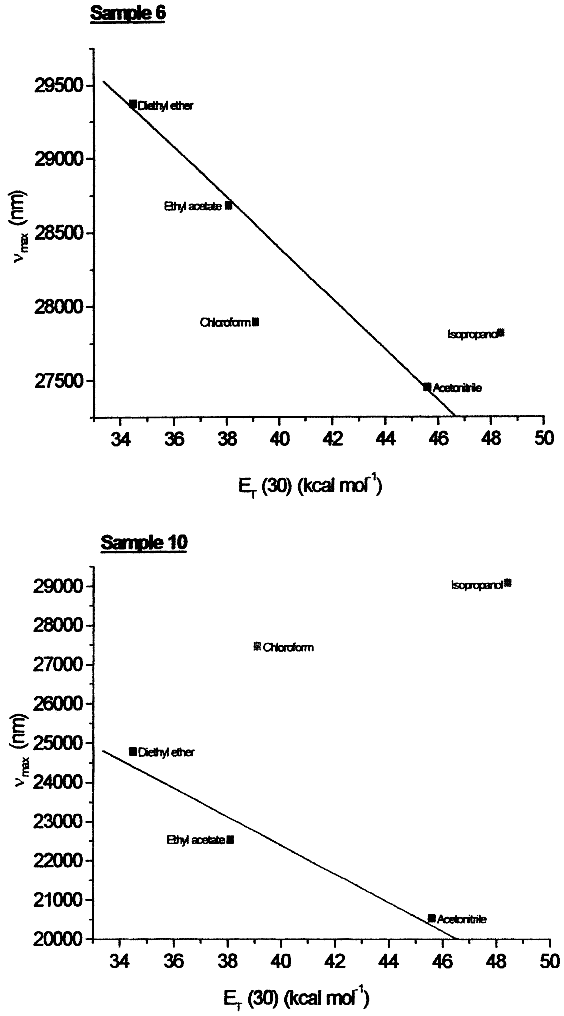


Fig. 2. Solvatochromic plots of wavenumber shift versus $E_T(30)$ for samples **6** and **10**.

Table 6. Although this photoreaction measures the reductive power of the co-synergists other factors play a role such as quenching and ground-state radical interactions. The quantum yields are generally low but overall the amides appear to photoreduce more effectively than the amines. Of the latter compound **8** is effective while for the former compound **3** is weak. A comparison of photoreductive power with amine/amide concentration is shown in Fig. 3. The amide compound **9** exhibits a linear relationship between quantum yield of photoreduction and concentration. Compound **8** exhibits a lower dependence and possibly stronger quenching effect on the excited triplet state of the thioxanthone initiator.

3.3. photocuring data

There is rarely agreement in the literature among the methodologies used to determine the efficiencies of initiators in their ability to photo-induce the curing of monomers or prepolymers. Several factors account for such anomalies and may be related to initiator–monomer interactions, light

Table 6
Photoreduction quantum yields of 2-propoxythioxanthone (4.2×10^{-4} M) in the presence of *p*-dimethylaminoesters and amides (10^{-3} M) in acetonitrile

Compound no.	Φ_r
Esters	
1	0.108
8	0.2096
10	0.1108
11	0.1517
Amides	
2	0.1544
3	0.064
4	0.2614
5	0.0794
6	0.1288
7	0.227
9	0.2775

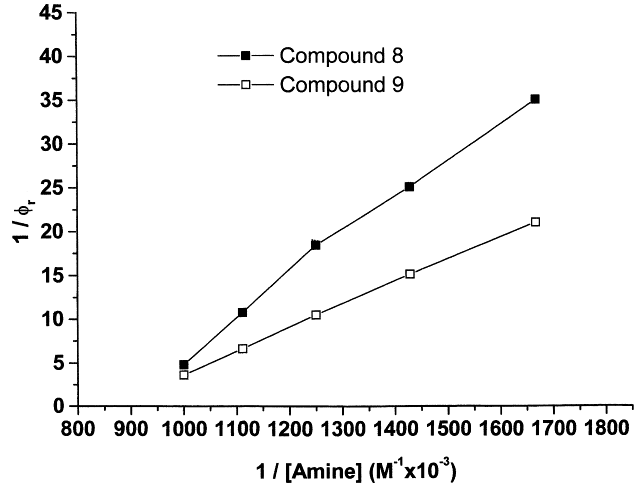


Fig. 3. Plot of inverse of photoreduction quantum yields vs. reciprocal of amine concentration for compounds (■) **8** and (□) **9**.

intensity and wavelength distribution, monomer viscosity effects on free radical migration/termination, physical or chemical measurements and the presence of oxygen. The results shown here are no exception but at least, provide a useful opportunity for comparison with scientific and technological data.

Pendulum hardness is a widely use industrial method of cure monitoring and probably one of the most quantitative methodologies in this regard. Most industrial methods tend to be very subjective. Pendulum hardness data is shown in Table 7. For the esters it is seen that substitution of the alkyl amine groups all give a beneficial effect in cure while for the amides compounds **5**, **7** and **9** have a lower effect than the esters especially the standard control (EPD). Compound **3** has a similar effect to that of EPD. The reduction in efficiency of the amides **5**, **7** and **9** from a structural point-of-view appear, to be associated with the bulky nature of the phenyl and piperazine substituents. Under the polychromatic irradiation source used in the photocuring, these functionalities may undergo some photolytic side reactions.

Table 7
Comparison of photocuring data: RTFTIR spectroscopy, photocalorimetry and pendulum hardness

Compound no.	Pendulum hardness	FTRTIR R_p (mol l s^{-1})	Photocalorimetry	
			$R_p \times 10^{-3}$ (mol dm^{-3} s^{-1})	Φ_m
Esters				
1	73–85	1.00	3.89	3.51
8	101–117	0.76	4.01	3.57
10	76–85	0.54	3.74	3.24
11	80–87	0.30	4.00	3.56
Amides				
2	83–88	1.00	0.60	0.59
3	73–83	2.18	4.10	3.78
4	76–87	0.97	3.74	3.76
5	56–67	0.43	3.85	3.53
6	80–94	0.60	3.90	3.86
7	67–71	0.57	3.82	3.53
9	60–64	0.62	3.82	3.34

The photocalorimetry data shown in Table 7 shows a close match to that for the pendulum hardness apart from the low activity of the primary amine, Compound **2** and the high activity of the secondary amine, compound **3**. In this work enthalpy only is measured a feature specific to the ensuing reactions in the calorimeter coupled with specific irradiation at 365 nm. In the latter case, photolysis of the amines/amides will be insignificant.

The RTFTIR data also shown in Table 7 shows a different pattern of behaviour. For amines the most active is the EPD control. However, the data for the amides apart from Compound **2** are similar to those for the pendulum hardness and photocalorimetry data. Here, compounds **5**, **7** and **9** exhibit low co-synergy with the 2-propoxythioxanthone initiator.

4. Conclusions

In this work three novel alkylamino ester and seven benzamide derivatives of *N*,*N*-dimethylaminobenzoic acid have been prepared and characterised. The esters exhibit longer wavelength absorptions than those of the amides and display a strong degree of charge-transfer in the lowest excited singlet state. The lowest excited singlet states of all the compounds are essentially $\pi\pi^*$ in character all providing some degree of photoreduction for the initiator 2-propoxythioxanthone. In general, the amides, apart from compound **3**, appear to be more effective in photoreduction than the esters.

The photocuring data for the different co-synergists shows some inconsistencies and appears to have no relationship with their photochemistry. This is in complete contrast with their primary initiator counterparts where correlations of photochemical and photophysical properties relates well to their photoactivities. Co-synergy is evident in all cases as 2-propoxythioxanthone is non-active alone [1–6]. The role of the amine co-synergist appears to be variable and dependent upon structure, electron donation and perhaps inherent photolytic stability. Overall, the amine esters appear to exhibit enhanced activity compared with the amides and in this way could be related to their greater degree of charge-transfer character enhancing co-synergy to form a more reactive triplet–exciplex with the thioxanthone initiator. However, the use of a bifunctional amine does not appear to offer any significant enhancements over that of the mono-substituted *N*,*N*-dimethylaminobenzoate group present in EPD.

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