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# SYNTHESIS AND PHOTOSTABILITY OF SOME NEW 1,8-NAPHTHALIMIDE DERIVATIVES AS COLOUR MONOMERS FOR FLUORESCENT POLYMERS

Polya M. Miladinova

*Organic Synthesis Department*
*University of Chemical Technology and Metallurgy*
*8 Kliment Ohridsky Blvd., Sofia1797, Bulgaria*
*ppolya@uctm.edu*

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## ABSTRACT

*Two new polymerizable 1,8-naphthalimide fluorophores containing a residue of a glycine or β-alanine in C-4 position of the naphthalimide ring have been synthesized. Their copolymers with methyl methacrylate were obtained. The colour coordinates of the prepared polymer have been determined. The photostability of the dyes and copolymers in dimethylformamide has been investigated and an increase in the photostability of dyes included in polymer with about 25 - 30 % was observed. The photostability of the polymer films obtained with copolymers and polymethyl methacrylate dyed "in mass" with the synthesized dyes has been determined by a change of colour coordinates and reflectance during the radiation with UV light. Results obtained lead to the conclusion that the dyes have good photostability and can be applied both as fluorescent dyes for dyeing of polymer materials "in mass" and as colour fluorescent monomers for obtaining of fluorescent polymers.*

*Keywords: 1,8-naphthalimide dyes, fluorescent polymers, photostability.*

## INTRODUCTION

Fluorescent polymers are widely used in various areas of industry and everyday life. They have an application as sensors for molecules, metal ions or acids [1 - 7], in cell and bio imagine [8 - 10], in textiles and polymer materials [1,11 - 13], and other fields [14 - 16].

One of the strategies for developing of fluorescent polymers is an incorporation of a fluorescent chromophore in the polymer chain, which can give photoluminescent properties to the final materials. The synthesis of fluorescent polymers can be accomplished by copolymerization of traditional monomers with fluorescent monomers (unsaturated fluorescent dyes). Among the different classes of the fluorescent dyes, the naphthalimide derivatives are attracting much attention due to their excellent fluorescent properties, high photo- and thermostability and possibility for application in different areas [17 - 37].

The introduction of some amino acids as glycine or alanine in the fluorescent monomers gives a possibility for obtaining of condensation products with different amino containing high molecular compounds. This can lead to changing of properties or activity of the compounds. For example, it was found that if some peptides are conjugated with second pharmacophore as 1,8-naphthalimide, containing alanine or glycine residue, the antitumor and antibacterial activity of the compound were increased [38 - 40].

This paper reports on the synthesis of novel 1,8-naphthalimide fluorophores, containing a residue of an unsaturated group and a residue of a glycine or β-alanine in C-4 position of the naphthalimide ring and the investigation of their possibility to form fluorescent polymers. The synthesized dyes and polymers obtained have been investigated for their photostability.

## EXPERIMENTAL

### Materials and methods

N-allyl-4-bromo-1,8-naphthalimide as a precursor for the synthesis of the target dyes has been synthesized according to a method described previously [34]. Glycine, β-alanine and azo-bis-iso-butyronitrile were products of Sigma Aldrich (Merck). Methyl methacrylate (MMA) was product of Fluka.

The melting point was recorded on a Büchi 535 apparatus (Switzerland). Thin-layer chromatography (TLC) was performed on silica gel plate (Merck, 60 F 254, 20 x 20 cm, 0.2 mm thickness). Electronic spectra were recorded on a Hewlett Packard 8452A UV/Vis spectrophotometer; IR spectra - on a Varian 660 instrument; $^1$H-NMR spectra - on DRX - 400 Brucker equipment and fluorescent spectra - on spectrometer FS-2 (Scinco).

The fluorescence quantum yields ($\Phi_F$) were measured relatively to Coumarin 6 ($\Phi_F$ = 0.78 in ethanol) as standards [35].

Photostability of the dyes (concentration $2.10^{-4}$ g mL$^{-1}$) and polymers (concentration $1.10^{-2}$ g mL$^{-1}$) in solution of dimethylformamide was studied by a Suntest CPS equipment (Heraeus) supplied with a Xenon lamp (Hanau, 1.1 kW, 765 W m$^{-2}$, wavelength λmax = 290 nm).

The copolymerization of the dye with MMA was performed according to a method described before [36].

Photostability of the dyes in the polymer films was studied by an exposure with a xenon light source on a test camera Q-SUN Xe-1 (Q-Lab, USA). The mode of operation of the test chamber is in accordance with BDS EN ISO 4892-2.

The polymer's films of copolymers and polymethyl methacrylate dyed "in mass" were prepared by effusion in plates of dissolved polymer in chloroform (concentration 0.06 g mL$^{-1}$). For the dyeing of polymethyl methacrylate (PMMA) 0.1 wt. % of the corresponding dye was added.

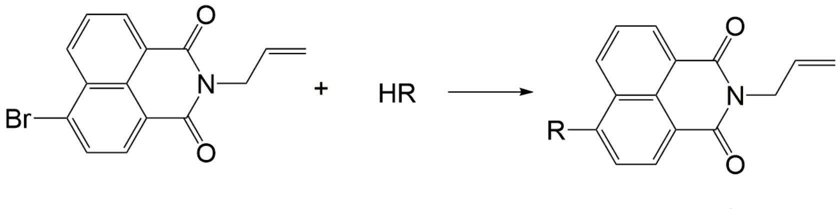
The colour characteristics of the polymers were recorded using ElektroPhysik ColorTest (Germany). The reflectance was measured with a Picogloss 560 MC (Erichsen) gloss meter.

### Synthesis of the dyes

N-allyl-4-bromo-1,8-naphthalimide (0.948 g, 0.003 mol) was dissolved in 25 mL dimethylformamide, then 0.003 mol of corresponding amino acid (0.225 g glycine or 0.267 g β-alanine) was added and the solutions were stirred for 6 h while boiling. The reactions were monitored by TLC (system hexane : acetone = 3 : 2 on silica gel). After cooling to 20°C, the reaction mixtures were poured into 50 mL water. The precipitates of the dyes obtained were filtered off, recrystallized with methanol and dried.

## RESULTS AND DISCUSSION

The synthetic route used for the preparation of 1,8-naphthalimide fluorophores (Dyes 1 and 2) is outlined in Scheme 1. The synthesized dyes were isolated and characterized.



Dyes 1 and 2

R = -NHCH$_2$COOH (Dye 1)

-NHCH$_2$CH$_2$COOH (Dye 2)

Scheme 1. Synthesis of the 1,8-naphthalimide fluorophores (Dyes 1 and 2).

**Characteristics of the dyes**

Dye 1: IR (KBr), cm$^{-1}$: 3450 (νNH); 3069 (νCH=CH); 2876 (νs $CH_2$); 1700 (νC=O); 1661 (νAr); 1588 (δNH); 1426 (δas $CH_2$); 1370 and 1234 (ν-C-N-); 778 (δAr); $^1$H NMR ($CDCl_3$, δ/ppm): 8.61-8.59 (d, 1H, ArH); 8.52-8.50 (d, 1H, ArH); 8.36-8.34 (d, 1H, ArH); 7.99-7.97 (d, 1H, ArH); 7.80-7.76 (t, 1H, ArH); 7.19 (s, 1H, -OH); 5.98-5.87 (m, 1H, -CH=); 5.28-5.10 (m, 3H, $=CH_2$, -NH); 4.74-4.72 (d, 2H, $-N\underline{CH_2}CH=CH_2$); 3.04 (s, 2H, $-NH\underline{CH_2}COOH$).

Dye 2: IR (KBr), cm$^{-1}$: 3500 (νNH); 3069 (νCH=CH); 2879 (νs $CH_2$); 1700 (νC=O); 1662 (νAr); 1589 (δNH); 1426 (δas $CH_2$); 1371 and 1234 (ν-C-N-); 778 (δAr); $^1$H NMR ($CDCl_3$, δ/ppm): 8.60-8.59 (d, 1H, ArH); 8.52-8.50 (d, 1H, ArH); 8.36-8.34 (d, 1H, ArH); 7.98-7.96 (d, 1H, ArH); 7.80-7.76 (t, 1H, ArH); 7.19 (s, 1H, -OH); 5.97-5.87 (m, 1H, -CH=); 5.28-5.10 (m, 2H, $=CH_2$, -NH); 4.74-4.72 (d, 2H, $-N\underline{CH_2}CH=CH_2$); 3.07-2.89 (t, 2H, $-NH\underline{CH_2}CH_2COOH$); 2.65-2.61 (t, 2H, $-NHCH_2\underline{CH_2}COOH$).

The photophysical properties of the dyes 1 and 2 were investigated in chloroform at a concentration of 1.10$^{-5}$ mol L$^{-1}$. Table 1 summarizes the basic spectral characteristics of the compounds: the values of absorption ($\lambda_A$) and fluorescence ($\lambda_F$) maxima, Stokes shift ($\nu_A$ - $\nu_F$) and the quantum yield of fluorescence ($\Phi_F$).

The absorption maximum of dyes 1 and 2 undergoes bathochromic shift in comparison with N-allyl-4-bromo-1,8-naphthalimide (344 nm) due to the electron donating ability of the imino group. The presence of the last one leads to the availability of a fluorescence in visible spectrum. The quantum yield of fluorescence $\Phi_F$ was calculated using equation (1) and Coumarin 6 ($\Phi_F$ = 0.78 in ethanol) as a standard [35], where $A_{ref}$, $S_{ref}$, $n_{ref}$ and $A_{sample}$, $S_{sample}$, $n_{sample}$ represent the absorbance at the excitation wavelength, the integrated emission band area and the solvent refractive index of the standard and the sample, respectively.

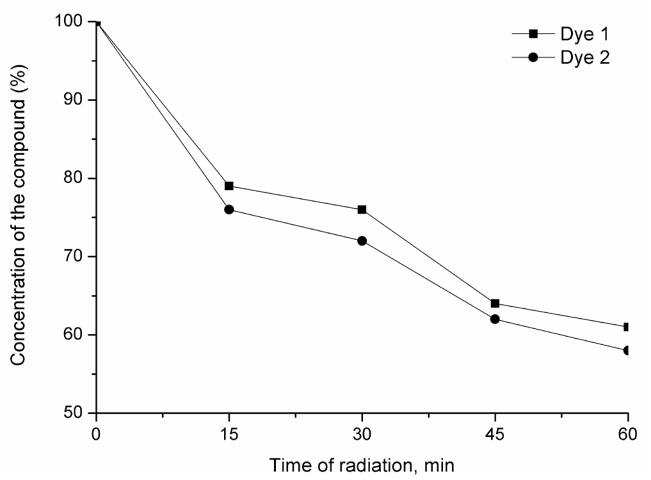


Fig. 1. The dependence of the compound's concentration (%) in solution on the time of radiation under UV light (min).

$$\Phi_F = \Phi_{ref}\left(\frac{S_{sample}}{S_{ref}}\right)\left(\frac{A_{ref}}{A_{sample}}\right)\left(\frac{n^2_{sample}}{n^2_{ref}}\right) \qquad (1)$$

**Photostability of the dyes**

Solutions of the dyes in dimethylformamide were subjected to radiation by UV light for 1 hour in Suntest equipment. The photodegradation of the dyes was monitored colourimetrically, i.e. the dependence of the concentration of the dye on the time of radiation was determined using the method of the standard calibration curve; the concentration of the compounds in nonradiated solutions was accepted to be 100 % (Fig. 1).

One can see that the concentration of the dyes in the end of radiation is 61 % for dye 1 and 58 % for dye 2. These values indicate a high photostability.

**Copolymerization with methyl methacrylate**

The copolymerization of methyl methacrylate with dyes 1 and 2 was performed according to the

Table 1. Characteristics of dyes.

| Compound | Yield, % | Rf$^a$ | MP, °C | $\lambda_A$, nm | $\lambda_F$, nm | $\nu_A$ - $\nu_F$, cm$^{-1}$ | $\Phi_F$$^b$ |
|---|---|---|---|---|---|---|---|
| 1 | 78 | 0.61 | 107 - 110 | 418 | 506 | 4161 | 0.643 |
| 2 | 84 | 0.58 | 122 - 124 | 420 | 506 | 4047 | 0.610 |

[a] *TLC (system hexane : acetone = 3 : 2 on silica gel);* [b] *$\Phi_F$ was measured relatively to Coumarin 6 ($\Phi_F$ = 0.78 in ethanol) [35]*

standard procedure with 0.1 wt. % of the dye [33]. After 8 h yellow-green, fluorescent polymers were obtained. They were precipitated with methanol two times. Polymers thus treated were coloured, which was an indication that the dyes were covalently bonded in polymer. The percentage of chemically bonded compounds was calculated by the method of the standard calibration curve; the concentration of the compounds in non-precipitated polymers was accepted to be 100 %. It was found that the chemically bonded dye in the copolymer is 63 % (for dye 1) and 65 % (for dye 2).

### Photostability of the dyes in polymers

To study the photostability of the compounds in the copolymers and in PMMA dyed "in mass", the solutions of the precipitated polymers were radiated with UV light under the same conditions used for the solutions of the pure compounds for 8 h. The process was monitored spectrophotometrically, as the concentration of the dyes before radiation was accepted to be 100 %. The data are presented in Fig. 2.

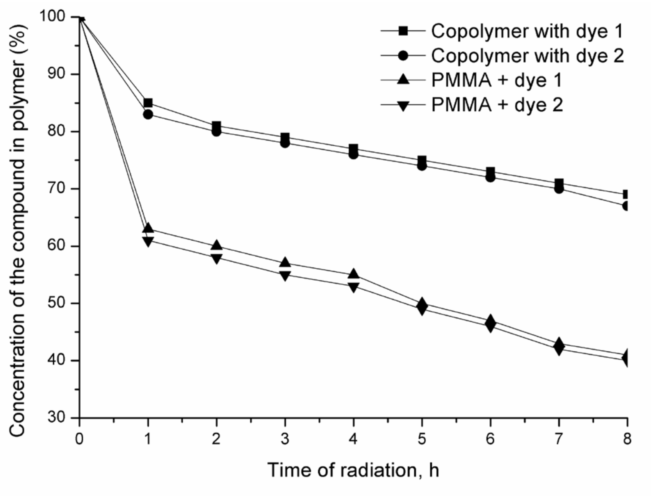


Fig. 2. The dependence of the compound's concentration in copolymer (%) and in PMMA dyed "in mass" on the time of radiation under UV light (h).

One can see from Fig. 2 that the photostability of the dyes is increased by 25 - 30 % when they are incorporated in the polymer chain.

The photostability of the dyes was determined also by investigation of the changes in colour characteristics and reflectance R of the copolymers and PMMA dyed "in mass" (Table 2). For this purpose, the polymer's films were radiated with UV light for 8 h. The colour characteristics and the reflectance of the samples were recorded every hour. The data for the lightness value L* during the radiation are presented in Table 3 and Table 4.

From the results in the Table 2, the colour of the polymer films obtained with the corresponding dyes is the same, i.e. the colour of the dyes does not change during the copolymerization, which is one of the main requirements for the chemical colouration of polymers. The reflectance values of the copolymers and PMMA coloured "in mass" are comparable.

Table 2. Colour characteristics (L*, a*, b* and ΔE) and reflectance (R) of the polymer films.

| Sample | L* | a* | b* | ΔE | R |
|---|---|---|---|---|---|
| PMMA | 94.51 | 0.85 | -7.08 | 8.99 | 113.4 |
| Copolymer with dye 1 | 95.80 | -7.58 | 6.26 | 10.4 | 102.0 |
| PMMA + dye 1 ("in mass") | 94.25 | -18.02 | 24.68 | 30.83 | 92.5 |
| Copolymer with dye 2 | 94.71 | -5.03 | 2.15 | 7.31 | 102.3 |
| PMMA + dye 2 ("in mass") | 95.00 | -7.37 | 6.07 | 10.48 | 95.9 |

Table 3. The data for the lightness value L* during the radiation with UV light.

| Sample | 0h | 1h | 2h | 3h | 4h | 5h | 6h | 7h | 8h |
|---|---|---|---|---|---|---|---|---|---|
| PMMA | 94.10 | 93.67 | 93.54 | 94.15 | 93.54 | 93.11 | 93.61 | 93.37 | 94.19 |
| Copolymer with dye 1 | 95.80 | 95.91 | 95.99 | 96.38 | 96.58 | 96.66 | 96.72 | 96.77 | 96.91 |
| PMMA + dye 1 (in mass) | 94.25 | 94.69 | 95.33 | 95.66 | 96.54 | 96.73 | 97.22 | 97.88 | 98.37 |
| Copolymer with dye 2 | 95.56 | 95.87 | 95.91 | 96.48 | 96.78 | 97.12 | 97.43 | 97.61 | 97.75 |
| PMMA + dye 2 (in mass) | 94.24 | 95.31 | 95.73 | 95.92 | 96.34 | 96.36 | 97.18 | 97.37 | 97.71 |

Table 4. The data for the reflectance R during the radiation with UV light.

| Sample | 0h | 1h | 2h | 3h | 4h | 5h | 6h | 7h | 8h |
|---|---|---|---|---|---|---|---|---|---|
| PMMA | 97.2 | 87.1 | 85.7 | 82 | 54.2 | 51.7 | 48.1 | 45.9 | 42.2 |
| Copolymer with dye 1 | 97.0 | 40.2 | 32.6 | 29.9 | 28.8 | 25.6 | 25.5 | 25.4 | 22.5 |
| PMMA + dye 1 (in mass) | 92.5 | 67.1 | 56.3 | 54.2 | 50.3 | 48.6 | 45.2 | 44.5 | 43.6 |
| Copolymer with dye 2 | 96.7 | 64.3 | 44.4 | 41.1 | 34.4 | 30 | 28.6 | 25.6 | 20.7 |
| PMMA + dye 2 (in mass) | 90.5 | 60.7 | 55.9 | 55.3 | 55 | 54.7 | 54.2 | 48.3 | 42.2 |

As can be seen from Table 3 and 4, the L* value of the reference sample (PMMA) remains stable for the entire radiation period and increases with 1 - 2 % for the copolymers and with 4 % for the PMMA dyed “in mass”. All simples show a significant decrease in the reflectance within the 1st hour. In the end of the 8th hour the reflectance decreases with 77 - 79 % for the copolymers and with 53 % for the PMMA dyed “in mass”. This is probably due to cross-linking and microcracks on the surface of the test body because of the intense radiation and moisture.

## CONCLUSIONS

Two new unsaturated 1,8-naphthalimide fluorophores containing residue of a glycine or β-alanine have been synthesized. The dyes are capable to copolymerize with methyl methacrylate and thus intensively coloured polymers have been obtained. The photostability of the dyes in solution and polymer was investigated. The colour characteristics and reflectance of polymer’s films of the copolymers and polymethyl methacrylate dyed “in mass” were determined. Results obtained lead to the conclusion that the dyes have good photostability which increases when they are incorporated in polymer chain.

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***Authors’ contributions****: All studies described in the manuscript are prepared by P.M.*

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