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Synthesis of new polymerizable 1,8-naphthalimide dyes containing a 2-hydroxyphenylbenzotriazole fragment

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Abstract

The synthesis of new 1,8-naphthalimide dyes, containing 2-hydroxyphenylbenzotriazole stabiliser fragment is reported. Two polymerizable dyes, a combination between 1,8-naphthalimide and 2-hydroxyphenylbenzotriazole, as well as four 1,8-naphthalimide intermediates were synthesised in good yields under variable reaction conditions. The ability of the dyes for co-polymerisation with vinyl monomers such as acrylonitrile was demonstrated. The percentage of the covalently bound dyes into the polymer chain and the co-polymers' both viscosity and molecular weight were determined. It was found that the monomeric dyes did not significantly affect the co-polymerisation process.

Keywords: Polymerizable fluorescent dyes containing stabiliser; 1,8-Naphthalimide; 2-Hydroxyphenylbenzotriazole; Co-polymerisation

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1. Introduction

The structural coloration of synthetic polymer materials gives a possibility of obtaining polymers with a colour stable to wet treatment and solvents [1–3]. The co-polymers of traditional monomers with some polymerizable fluorescent units display intensive fluorescence [4]. Thus, using appropriate fluorophores, polymers of a different fluorescent colour can be obtained. On the other hand photo stability of polymers is of great importance for their use. The covalent bonding of the stabiliser to the polymer chain provided a good stability to solvents and migration, improving their environmental behaviour [5]. We have reported before on the synthesis of different polymerizable stabilisers and their co-polymerisation with styrene and methyl methacrylate [6–10]. In our earlier papers [11,12] we demonstrated the possibility for "one-step" coloration and stabilisation of polystyrene and polyacrylonitrile, where a fluorescent dye and a stabiliser were combined in one molecule.

It is well know that fluorescent 1,8-naphthalimide dyes have a bright colour and possess good dyeability with synthetic polymers. They have found applications in sun energy collectors [13], laser active media [14,15], potential photosensitive biologically active units [16], also as fluorescent

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markers in biology [17], and in medicine [18,19]. Recently they have been examined in liquid crystals for utilisation in electro-optical devices [20,21]. In previous papers we have reported on the synthesis and the properties of 4-amino-1,8-naphthalimide derivatives as dyes for polymers. The dyes possess an intensive yellow–green fluorescence and very good photostability [22–25]. Due to the presence of a polymerizable group the dyes can undergo polymerisation with some commercial monomers, allowing coloured co-polymers with an intensive fluorescence to be obtained [26,27].

Bearing in mind these investigations it was of interest to synthesise some new naphthalimide derivatives, where a polymerizable group and a stabiliser were to be combined in the dye molecule. Thus, this was the object of the present study.

2. Experimental

2.1. Materials

The intermediates 4-nitro-*N*-allyl-1,8-naphthalimide **5a** and 4-bromo-*N*-allyl-1,8-naphthalimide **5b**, the starting materials 4-nitro-1,8-naphthalic anhydride **1a** and 4-bromo-1,8-naphthalic anhydride **1b** as well as 2-(2-hydroxy-phenyl)-5-amino-2*H*-benzotriazolå **2** were synthesised according to procedures described before [10,21–23].

Acrylonitrile (AN) was distilled before use. Dibenzoylperoxide (DBP) (Fluka 99.6%) was recrystallised from chloroform. *N,N*-Dimethylformamide (DMF) (Merck) for synthesis and of a spectroscopy grade was used.

2.2. Analysis

FT-IR spectra were recorded on a Bruker IFS-113 spectrometer at 2 cm^{-1} resolution using KBr discs. ^{1}H-NMR spectra were recorded on a Bruker spectrometer, operating at 250 MHz in $CDCl_3$ (chemical shifts are given as δ in ppm). UV/Vis spectra were recorded on a Hewlett Packard 8452A UV/Vis spectrophotometer with 2 nm resolution, at room temperature in DMF. The reaction course and purity of the final products were followed by TLC on silica gel (Fluka F_{60}254 20×20, 0.2 mm). The melting points were determined by means of a Kofler melting point microscope.

2.3. Synthesis of intermediates and final dyes

2.3.1. Synthesis of 6-allylamino-2-[2-(2-hydroxy-phenyl)-2H-benzotriazol-5-yl]-benzo[de]isoquinoline-1,3-dione 4

To a solution of 2-[2-(2-hydroxy-phenyl)-2*H*-benzotriazol-5-yl]-6-nitro-benzo[*de*]isoquinoline-1,3-dione **3a** (2.26 g, 0.005 mol) in 30 ml of DMF allylamine (0.29 g, 0.005 mol) was added at room temperature. After 24 h (TLC control in a solvent system *n*-heptane:acetone = 1:1), the resulting solution was poured into 300 ml of water. The precipitate was filtered off, washed with water and dried. Final 6-allylamino-2-[2-(2-hydroxy-phenyl)-2*H*-benzotriazol-5-yl]-benzo[*de*]isoquinoline-1,3-dione **4** was obtained as yellow crystals in 87% yield.

2.3.2. Synthesis of 2-allyl-6-[2-(2-hydroxy-phenyl)-2H-benzotriazol-5-ylamino]-benzo[de]isoquinoline-1,3-dione 6

To a solution of 2-(2-hydroxy-phenyl)-5-amino-2*H*-benzotriazole **2** (1.13 g, 0.005 mol) in 40 ml of glacial acetic acid were added 0.75 g (0.005 mol) of sodium acetate and 1.58 g (0.005 mol) of 4-bromo-*N*-allyl-1,8-naphthalimide **5b**. The resulting mixture was stirred for 4 h at 120 °C (TLC control in a solvent system *n*-heptane:acetone = 1:1) and then after cooling the precipitated product was filtered off, washed with water and dried. Final 2-allyl-6-[2-(2-hydroxy-phenyl)-2*H*-benzotriazol-5-ylamino]-benzo[*de*]isoquinoline-1,3-dione **6** was obtained as yellow crystals in 44% yield.

2.3.3. Synthesis of intermediate 2-[2-(2-hydroxy-phenyl)-2H-benzotriazol-5-yl]-6-nitro-benzo[de]isoquinoline-1,3-dione 3a

A solution of 2-(2-hydroxy-phenyl)-5-amino-2*H*-benzotriazole **2** (2.26 g, 0.01 mol) and 4-nitro-1,8-naphthalic anhydride **1a** (2.43 g, 0.01 mol) in 80 ml of glacial acetic acid was stirred under refluxing for 12 h (TLC control in a solvent system *n*-heptane:acetone = 1:1). The crude product which precipitated on cooling was treated with 100 ml of

5% aqueous sodium hydroxide to give after filtration, washing with water and drying 3.07 g (68% yield) of 2-[2-(2-hydroxy-phenyl)-2*H*-benzotriazol-5-yl]-6-nitro-benzo[*de*]isoquinoline-1,3-dione **3a** as pale yellow-brown crystals.

2.3.4. *Synthesis of intermediate 2-[2-(2-hydroxy-phenyl)-2*H*-benzotriazol-5-yl]-6-bromo-benzo-[*de*]isoquinoline-1,3-dione **3b***

A solution of 2-(2-hydroxy-phenyl)-5-amino-2*H*-benzotriazole **2** (2.26 g, 0.01 mol) and 4-bromo-1,8-naphthalic anhydride **1b** (2.77 g, 0.01 mol) in 80 ml of glacial acetic acid was stirred under refluxing for 18 h (TLC control in a solvent system *n*-heptane:acetone = 1:1). The crude product which precipitated on cooling was treated with 100 ml of 5% aqueous sodium hydroxide to give after filtration, washing with water and drying 2.96 g (61% yield) of 2-[2-(2-hydroxy-phenyl)-2*H*-benzotriazol-5-yl]-6-bromo-benzo[*de*]isoquinoline-1,3-dione **3b** as pale yellow-brown crystals.

2.4. *Synthesis of polymers*

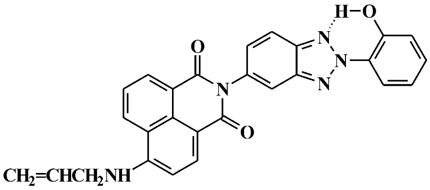
The radical co-polymerisation of AN with the dyes **4** and **6** was carried out in vials, purged with pure nitrogen before use. The co-polymerisation was conducted under conditions used with other similar 1,8-naphthalmide derivatives [12,28]. The concentration of DBP and the dyes were 1 wt.% initial concentration with respect to the AN monomer. The vials were treated in a thermostat for 12 h at 70 °C. The side-group co-polymers thus obtained were re-precipitated several times with methanol from DMF in order to remove the non-interacted dye monomers. The co-polymers were dried to a constant weight in vacuum at 40 °C. All spectrophotometric measurements were carried out with precipitated polymers.

2.5. *Determination of polymer molecular weights*

The molecular weights of polyacrylonitrile (PAN) and poly(AN-co-dye)s were determined by measuring the limiting viscosity number [η] in DMF solution, using an Ubbelohde viscosimeter at 25 °C. Mark-Houwink equation: $[\eta] = KM_v^a$ was used for determination of the average viscosimetric molecular weight (M_v) [29]. The respective constants for PAN are $K = 1.75 \times 10^{-3}$ and $a = 0.66$ [30].

3. Result and discussion

Following the aim of the present study we synthesised polymerizable 1,8-naphthalimide dyes containing a 2-hydroxyphenylbenzotriazole fragment and a reactivity allyl group in one molecule. The synthesised compounds are represented by Formula 1 and Formula 2.



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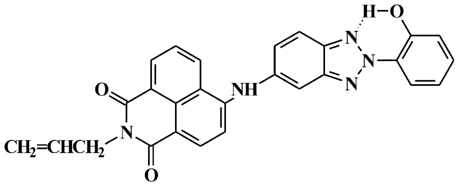
Formula 1.



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Formula 2.

3.1. *Synthesis of dyes*

3.1.1. *Synthesis of 6-allylamino-2-[2-(2-hydroxy-phenyl)-2*H*-benzotriazol-5-yl]-benzo[*de*]isoquinoline-1,3-dione **4***

The target 6-allylamino-2-[2-(2-hydroxy-phenyl)-2*H*-benzotriazol-5-yl]-benzo[*de*]isoquinoline-1,3-dione **4** was synthesised in just two steps following Scheme 1.

First, 4-substituted-1,8-naphthalic anhydrides **1a,b** were converted into 2-[2-(2-hydroxy-phenyl)-2*H*-benzotriazol-5-yl]-6-substituted-benzo[*de*]isoquinoline-1,3-diones **3a,b** by reaction with 2-(2-hydroxy-phenyl)-5-amino-2*H*-benzotriazol **2**. In order to obtain 1,8-naphthalimide dye and functionalise it with polymerizable group, the intermediates **3a,b** thus synthesised were reacted with allylamine.

Both synthetic steps (Scheme 1) were carried out (TLC control) in 1:1 molar ratio under different reaction conditions (solvents, reaction temperature and reaction time).

Different solvents (DMF, glacial acetic acid and toluene) were tested as a reaction medium. The best results in the synthesis of intermediates **3a,b** were obtained when the reaction was carried out in the medium of glacial acetic acid. The data obtained also showed that the strong electron-accepting nitro group considerably improve reaction ability of the starting 4-nitro-1,8-naphtalic anhydride **1a** (intermediate **3a**).

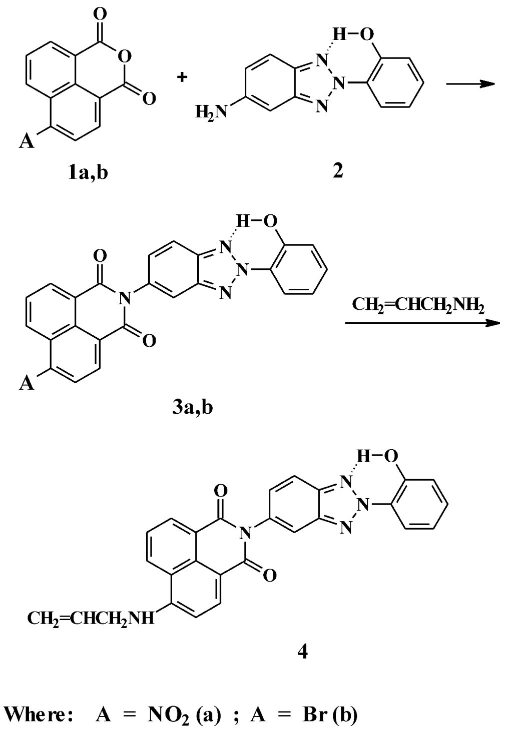
The results obtained for the synthesis of the final dye 6-allylamino-2-[2-(2-hydroxy-phenyl)-2*H*-benzotriazol-5-yl]-benzo[*de*]isoquinoline-1,3-dione **4** were good enough when at room temperature DMF was used as a reaction medium (interaction of 2-[2-(2-hydroxy-phenyl)-2*H*-benzotriazol-5-yl]-6-nitro-benzo[*de*]isoquinoline-1,3-dione **3a** with allylamine). The target dye **4** was not obtained when the intermediate 2-[2-(2-hydroxy-phenyl)-2*H*-benzotriazol-5-yl]-6-bromo-benzo[*de*]isoquinoline-1,3-dione **3b** was reacted with allylamine under these conditions.

3.1.2. Synthesis of 2-allyl-6-[2-(2-hydroxy-phenyl)-2H-benzotriazol-5-ylamino]-benzo[de]isoquinoline-1,3-dione 6

The synthesis of 2-allyl-6-[2-(2-hydroxy-phenyl)-2*H*-benzotriazol-5-ylamino]-benzo[*de*]isoquinoline-1,3-dione **6** was in two steps as well, but just in the reverse order following Scheme 2.

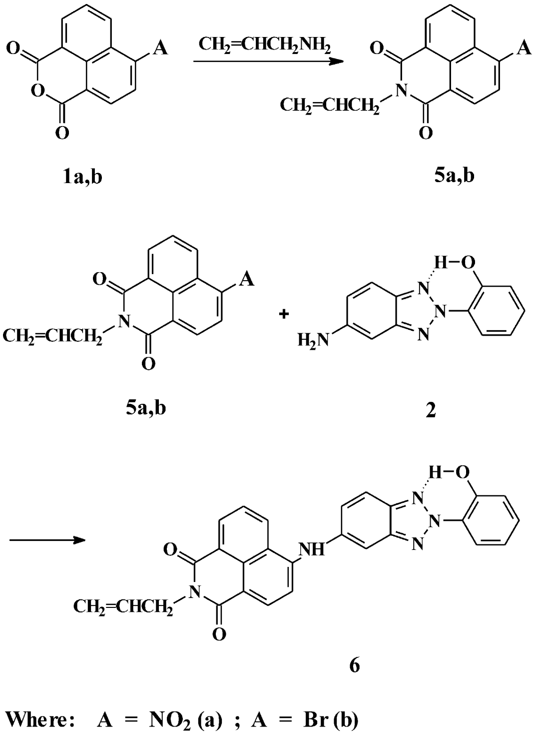
The intermediates 4-nitro-*N*-allyl-1,8-naphthalimide **5a** and 4-bromo-*N*-allyl-1,8-naphthalimide **5b** were synthesised using starting compounds **1a,b** as described by us before [22,23].

The target 2-allyl-6-[2-(2-hydroxy-phenyl)-2*H*-benzotriazol-5-ylamino]-benzo[*de*]isoquinoline-1,3-dione **6** was obtained by a nucleophilic substitution



Where: A = NO_2 (a) ; A = Br (b)

Scheme 1.



Where: A = NO_2 (a) ; A = Br (b)

Scheme 2.

of the bromine atom in the intermediate 4-bromo-*N*-allyl-1,8-naphthalimide **5b.** The reaction of the latter with 2-(2-hydroxy-phenyl)-5-amino-2*H*-benzotriazol **2** was carried out in 1:1 molar ratio in glacial acetic acid at 120 °C. The nucleophilic substitution of the nitro group in the intermediate 4-nitro-*N*-allyl-1,8-naphthalimide **5a** was not take place neither in glacial acetic acid at 120 °C nor in DMF at room temperature and the final product (dye **6**) was not obtained under these conditions. Better results were obtained when the reaction was carried out in DMF at higher temperatures, but the yield of the desired dye **6** was lower than those when 4-bromo-*N*-allyl-1,8-naphthalimide **5b** was used as an intermediate.

The synthesis of the desired compounds was monitored by TLC on silica gel and the intermediates **3a,b** as well as the final products **4** and **6** were characterized by their melting points, TLC R_f values and UV/Vis spectra (Table 1) and identified by elemental analysis, IR and ^{1}H-NMR spectra (Tables 2 and 3).

Data presented in Table 3 confirmed the structures of the synthesised dyes **4** and **6**.

Table 1
Yields and characterisation data for intermediates **3a**,**b** and dyes **4** and **6**

Compound	Yield %	M.p. °C	R_f[a]	λ_1[b] nm	log ϵ	λ_2[c] nm	log ϵ
3a	68	>260	0.52	334	4.19		
3b	61	>260	0.48	336	4.20		
4	87	>260	0.44	340	4.23	424	4.14
6	44	>260	0.42	346	4.21	428	4.12

[a] Eluant system *n*-heptane:acetone = 1:1.
[b] Absorption band for the benzotriazole fragment.
[c] Absorption band for the 1,8-naphthalimide fragment.

Table 2
Identification of intermediates **3a**,**b**

Compound	Analysis%	FT-IR cm^{-1}	^{1}H-NMR δ ppm
3a	$C_{24}H_{13}N_5O_5$ MW 451.39 Calcd. C = 63.86 H = 2.90 N = 15.52 Found C = 64.12 H = 2.85 N = 15.61	3420 (νOH); 3078 (ν = CH); 1712 (ν^{as}C = O); 1673 (ν^{s}C = O); 1596 (νC = C); 1528 ($\nu^{as}NO_2$); 1369 ($\nu^{s}NO_2$); 1344 (νN-C-N); 1244 (νC-O-C).	8.68 (*m*, 1H, naphthalimide 7-H); 8.24 (*m*, 4H, naphthalimide 4-H, 5-H and 9-H, benzotriazole. 4-H); 7.95 (*m*, 3H, naphthalimide 8-H, benzotriazole 6-H and 7-H); 7.41 (*m*, 2H, phenyl 4-H and 6-H); 7.18 (*m*, 2H, phenyl 3-H and 5-H); 4.88 (*br.s*, 1H, OH).
3b	$C_{24}H_{13}BrN_4O_3$ MW 485.29 Calcd. C = 59.40 H = 2.70 N = 11.55 Found C = 59.18 H = 2.77 N = 11.66	3416 (νOH); 3081 (ν = CH); 1707 (ν^{as}C = O); 1669 (ν^{s}C = O); 1584 (νC = C); 1346 (νN-C-N); 1252 (νC-O-C).	8.32 (*m*, 3H, naphthalimide 7-H and 9-H, benzotriazole 4-H); 7.85 (*m*, 5H, naphthalimide 4-H, 5-H and 8-H, benzotriazole 6-H and 7-H); 7.28 (*m*, 2H, phenyl 4-H and 6-H); 7.09 (*m*, 2H, phenyl 3-H and 5-H); 4.19 (*br.s*, 1H, OH).

3.2. Polymerisation and characterisation of co-polymers

N,N-Dimethylformamide is one of the best solvents for the polyacrylonitrile (PAN) and it is used extensively. Recently the kinetic of the co-polymerisation of acrylonitrile (AN) monomers with some 1,8-naphthalimide derivatives in DMF solution was investigated in details [26,28]. In all cases the participation of monomeric 1,8-naphthalimide derivatives retarded feebly the process of the co-polymerisation.

In this study the ability of the synthesised dyes for co-polymerisation with vinyl monomers such as AN was investigated. The polymerisation reactions of dyes **4** and **6** with AN were conducted in sealed vials using dry DMF as a solvent and DBP as an initiator.

The effect of the co-polymer molecular weights has been of great importance for their practical utilisation. To determine M_v we used the viscosimetric method. Because of the low percentage of the dyes incorporated into the polymer chain (1.0

Table 3
Identification of dyes **4** and **6**

Dye	Analysis%	FT-IR cm^{-1}	^{1}H-NMR δ ppm
4	$C_{27}H_{19}N_5O_3$ MW 461.47 Calcd. C = 70.27 H = 4.15 N = 15.18 Found C = 70.60 H = 4.21 N = 15.06	3454 (νOH); 3369 (νNH); 3080 (ν = CH); 2922 (νCH); 1696 (ν^{as}C = O); 1657 (ν^{s}C = O); 1584 (νC = C); 1365 (νN-C-N); 1242 (νC-O-C).	8.36 (*m*, 1H, benzotriazole 4-H); 8.01 (*m*, 4H, naphthalimide 4-H, 7-H, 9-H and benzotriazole 7-H); 7.66 (*t*, 1H, *J* = 8 Hz, NH); 7.40 (*m*, 4H, naphthalimide 8-H, benzotriazole 6-H, phenyl 4-H and 6-H); 7.13 (*m*, 3H, naphthalimide 5-H, phenyl 3-H and 5-H); 6.02 (*m*, 1H, allyl =CH); 5.31 (*m*, 2H, allyl =CH_2); 4.02 (*d*, 2H, *J* = 5 Hz, allyl NCH_2); 3.51 (*br.s*, 1H, OH).
6	$C_{27}H_{19}N_5O_3$ MW 461.47 Calcd. C = 70.27 H = 4.15 N = 15.18 Found C = 69.99 H = 4.08 N = 15.29	3460 (νOH); 3355 (νNH); 3074 (ν = CH); 2925 (νCH); 1704 (ν^{as}C = O); 1665 (ν^{s}C = O); 1597 (νC = C); 1349 (νN-C-N); 1226 (νC-O-C).	8.26 (*s*, 1H, NH); 7.93 (*m*, 3H, naphthalimide 4-H, 7-H and 9-H); 7.64 (*m*, 2H, naphthalimide 8-H and benzotriazole 7-H); 7.32 (*m*, 3H, benzotriazole 4-H, phenyl 4-H and 6-H); 7.06 (*m*, 4H, naphthalimide 5-H, benzotriazole 6-H, phenyl 3-H and 5-H); 6.26 (*m*, 1H, allyl =CH); 5.54 (*m*, 2H, allyl =CH_2); 4.32 (*d*, 2H, *J* = 6 Hz, allyl NCH_2); 3.93 (*br.s*, 1H, OH).

Table 4
Yields and characterisation data for poly(AN-co-dye)s

Polymer	Yield %	Chemically bonded dye wt.%	λ_{max}[a] nm	[η] cm^3 g^{-1}	M_v
PAN	84			2.00	43,000
poly(AN-co-dye **4**)	71	0.80	428	1.89	39,500
poly(AN-co-dye **6**)	68	0.75	430	1.87	38,800

[a] Absorption band for the 1,8-naphthalimide fragment.

wt.%) towards acrylonitrile we had a reason to use the values *K* and *a* for homopolyacrylonitrile in the Mark-Houwink equation. The average viscosimetric molecular weights M_v determined for poly(AN-co-dye**6**) and poly(AN-co-dye**4**) were 38,800 and 39,500, respectively, and 43,000 for the homopolymer (Table 4). These results showed that dyes **4** and **6** did not affect significantly the co-polymers' molecular weight, when compared to that of the homopolymer.

The UV-Vis absorption spectra of the co-polymers showed similar absorption maxima as those of the monomeric dyes (Table 4). This is an indication that no changes occurred in the basic chromophoric systems of dyes **4** and **6**, neither during the polymerisation, nor as a result of their incorporation to the polymer chain. On this basis, we decided to use the method of the standard curve for spectrophotometric determination the content of a chemically bound dye in the polymer. It was estimated that 0.80 wt.% of dye **4** and 0.75 wt.% of dye **6** were chemically bonded into the re-precipitated polymer macromolecules. Considering that, during the precipitation, a part of the reacted dyes was removed in the low-molecular weight fraction, this percentage is satisfactory. Both dyes **4** and **6** showed different activity due to the position of the allyl groups in the chromophoric systems. Nevertheless more detailed investigations on the polymerisation process are necessary and they will be the object of our future study.

4. Conclusion

In this paper, we have presented the synthesis of new dyes, a combination of a stabiliser and a polymerizable group in the molecule of the 1,8-naphthalimide, and their co-polymerisation with AN. The monomeric dyes participated actively in the co-polymerisation and 0.75–0.80 wt.% of them was covalently bound into the polymer chain. They did not affect significantly the co-polymers' viscosity and molecular weight. On the basis of the results obtained it can be assumed that the new dyes are suitable for coloration of PAN. Investigations on the photo stability of the coloured polymers are the object of our future study.

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