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Chromophore functionalised maleimide copolymers with high poling stabilities of the nonlinear optical effect at elevated temperature

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Abstract

Nonlinear optical polymers with high glass transition temperatures were prepared by covalent functionalisation of polymaleimide copolymers with hydroxyalkyl chromophores via the Mitsunobu reaction. The polymaleimides were obtained by radical polymerisation of maleimide with 4-phenylstyrene (PM1), indene (PM2) or 1-adamantylmethacrylate (PM3). Glass transition temperatures in the range of 222–288°C were obtained. Thin spincoated films of the polymers were corona-poled and analysed by second-harmonic generation; second-order susceptibility values (d_{33}) up to 16 pm/V were obtained. Poled order stability measurements over periods of more than 200 h resulted in 72–90% of remaining NLO-response at 125°C. © 2001 Elsevier Science Ltd. All rights reserved.

Keywords: Polymaleimide; Nonlinear optics; Stability

1. Introduction

Second-order nonlinear optical (NLO) polymers are of considerable interest for the development of high-speed modulators and switches. Several important properties are required to be useful in applications for devices, e.g. large optical nonlinearities, low optical loss and stability of the NLO-response in function of time at prolonged elevated temperatures [1]. Network formation or crosslinking during poling is one of the approaches to solve the problem of poled-order stability [2,3]. Another approach is the synthesis of high glass transition (T_g) polymers, such as polyimides, that combine high NLO properties with good thermal stability [4–10]. Maleimide based polymers functionalised with chromophores also result in high T_g materials which, depending on their composition, give access to the existence of high thermal stability of the NLO-effect [11–13]. So far the latter have been prepared by a polymer analogous reaction of an amino functionalised chromophore and a poly(maleic anhydride) precursor copolymer, which gives formation of a poly(amic acid) copolymer that by reaction with acetic anhydride/pyridine under smooth conditions results in the respective maleimide copolymer. As maleic anhydride copolymers are sensitive to moisture, the polymers should be isolated under inert atmosphere and afterwards transformed into the respective chromophore functionalised polymaleimides by a two step reaction with aminoalkyl chromophore, which itself is difficult to purify and can give rise to irregularities in the copolymer structure. To avoid this harsh procedure, the aim of our investigation is the synthesis of maleimide copolymers with 4-phenylstyrene, indene and adamantyl methacrylate, respectively. These precursor polymers will then be functionalised with hydroxyalkyl chromophores in the presence of triphenylphosphine and diethylazodicarboxylate under Mitsunobu conditions [14]. In this way copolymers with a varied concentration of chromophore functionalised maleimide units may be obtained which will result in copolymers with different glass transition temperatures and high second-order nonlinearities.

2. Experimental part

2.1. Materials and instrumentation

All reagents were purchased from Aldrich Chemical Co. and Acros Organics. Maleimide and 2,2′-azobisisobutyronitrile were purified by crystallisation from ethylacetate and methanol, respectively. Reagent grade solvents were dried when necessary and purified by distillation.

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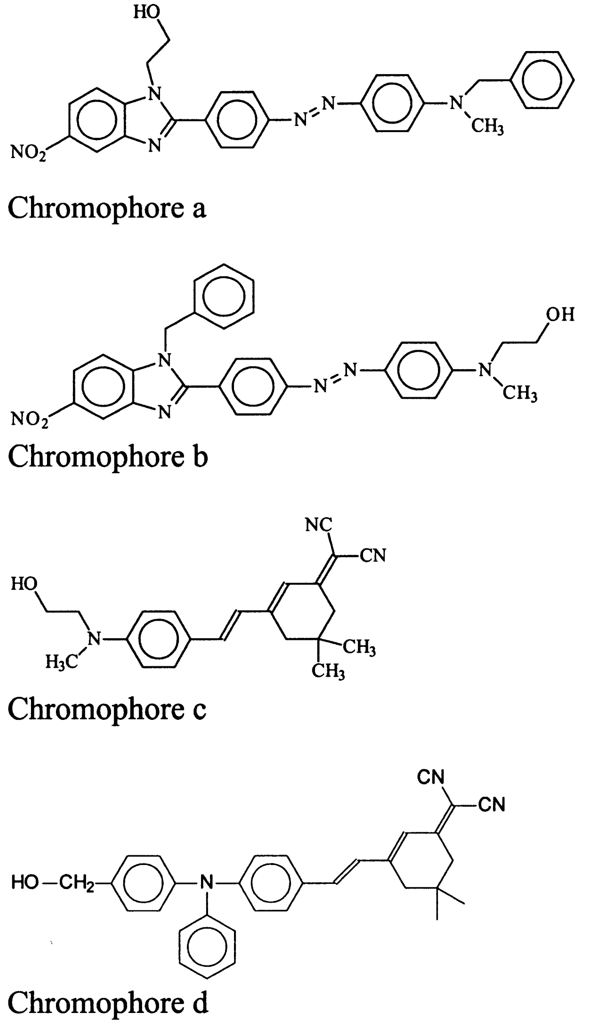


Chromophore a

Chromophore b

Chromophore c

Chromophore d

Fig. 1. Structures of chromophores a–d.

The glass transition and decomposition temperature were measured with a DSC-7 apparatus from Perkin–Elmer with a heating range of 20°C/min; typically the second run was taken for measuring T_g. The decomposition temperatures are higher than 320°C and could not be detected.

Gel permeation chromatography (GPC) measurements were done with a Waters apparatus with a tunable absorbance detector and a differential refractometer, in tetrahydrofuran (THF) as an eluent towards polystyrene standards. ^{1}H NMR measurements were done with a Bruker 300 MHz apparatus.

2.2. Second harmonic generation measurements

Thin films were obtained by spincoating a solution of the chromophore functionalised copolymers in cyclohexanone onto ITO substrates. The spincoated films were carefully dried under vacuum for at least 48 h at a temperature of about 10°C below the boiling point of the spincoating solvent. Subsequently they were corona-poled at a temperature approximately 10°C below T_g. The corona-poling set-up consisted of a thin wire, positioned 1 cm above the polymer film that was charged to 4–8 kV. The samples were poled for 30 min which gave best results in terms of SHG-efficiency. The second-harmonic coefficient d_{33} was measured using the standard Maker-fringe method [15]. A quartz crystal was used as a reference ($d_{11} = 0.3$ pm/V) and measurements were done at a fundamental wavelength of 1064 nm. Before each measurement, deposited charges from corona poling were wiped from the surface with methanol. The thermal stability of the NLO response was investigated by heating the corona-poled polymer films to 125°C and following the normalised second-harmonic coefficient $d_{33}(t)/d_{33}(t = 0)$ as a function of time, where $d_{33}(t)$ and $d_{33}(t = 0)$ represent the second-harmonic coefficient at times t and 0, respectively.

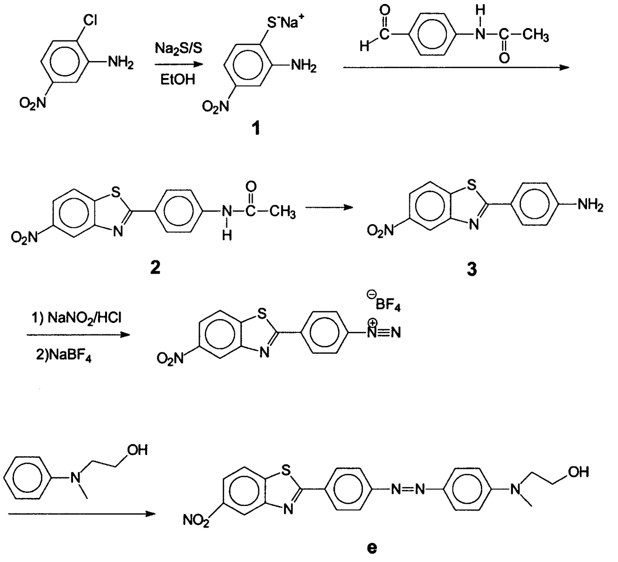


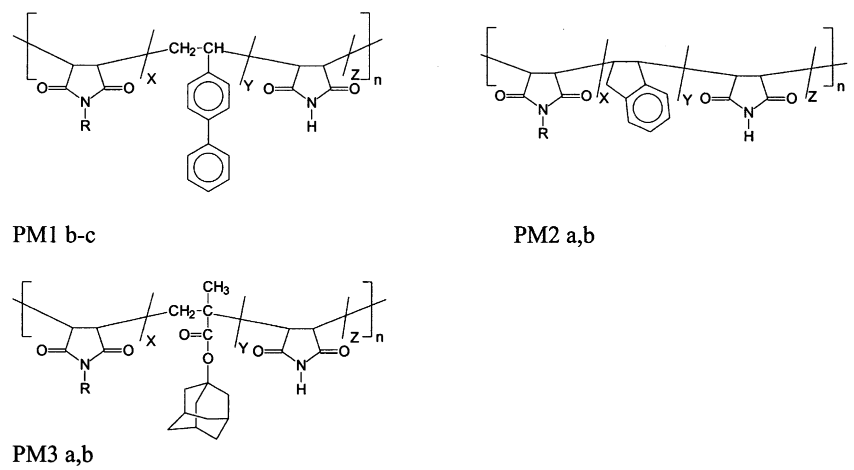
Fig. 2. Synthesis of chromophore e.

2.3. Synthesis of chromophores a–d (Fig. 1)

Chromophore a: 1-(2-hydroxyethyl)-2-[4-[4-(*N*-benzyl-*N*-methylamino)phenylazo]phenyl]-5-nitrobenzimidazole, mp 202°C. The procedure as described in Ref. [12] was followed. ^{1}H NMR (DMSO-d_6, ppm): $\delta = 3.19$ (s; 3H), 3.80 (m; 2H), 4.49 (t; 2H), 4.76 (s; 2H), 5.1 (t; 1H), 6.90 (d; 2H), 7.25–7.38 (m; 5H), 7.82 (d; 2H), 7.92 (m; 3H), 8.08 (d; 2H), 8.21 (dd; 1H), 8.59 (d,1H).

Chromophore b: 1-benzyl-2-[4-[4-(*N*-ethyl-*N*(2-hydroxyethyl) amino) phenylazo]phenyl]-5-nitrobenzimidazole, mp 189.6–190.7°C. The synthesis is described in a previous article [15]. ^{1}H NMR (DMSO-d_6, ppm): $\delta = 1.15$ (s; 3H), 3.53–3.61 (m; 6H), 4.83 (t; 1H), 5.77 (s; 2H), 6.85 (d; 2H), 7.03–7.26 (m; 5H), 7.78 (d; 1H) 7.79 (d; 2H) 7.88 (d; 2H) 7.92 (d; 2H), 8.2 (dd; 1H), 8.64 (d; 1H).

Chromophore c: Following the procedure described in Ref. [16]; mp 143.5–143.7°C. ^{1}H NMR (DMSO-d_6, ppm):



PM1 b-c

PM2 a,b

PM3 a,b

Fig. 3. Structures of functionalised copolymers PM1b–e, PM2a,b and PM3a,b (code a–e refers to the respective hydroxyalkyl chromophores a–e).

$\delta = 1.01$ (s; 6H), 2.52 (m; 4H), 3.01 (s; 3H), 3.47 (t; 2H), 3.57 (m; 2H), 4.71 (t; 2H), 6.72 (s; 1H), 6.73 (d; 2H), 7.10 (d; 1H), 7.23 (d; 1H), 7.53 (d; 2H).

Chromophore d: Ref. [17], mp 181.2°C. ^{1}H NMR (DMSO-d_6, ppm): $\delta = 1.1$ (s; 6H), 1.7 (s; 1H), 2.4 (s; 2H), 2.6 (s; 2H), 4.7 (s; 2H), 6.8 (s; 1H), 6.9 (d; 1H), 7.0–7.4 (m; 14H).

2.4. Synthesis of chromophore e (Fig. 2)

Thiophenolate 1: To a warm solution of 79.5 g (0.46 mol) of 2-chloro-5-nitroaniline in 500 ml of ethanol, a solution of 115 g (0.48 mol) of sodium sulfide monohydrate and 18 g of sulfur in 200 ml of ethanol was added. After 1 h of reflux and cooling, thiophenolate 1 was isolated.

2-(4-acetamidophenyl)-5-nitrothiobenzimidazole 2: 10 g (0.055 mol) of 1 and 9.3 g (0.057 mol) of 4-acetamidobenzaldehyde in 70 ml of acetic acid are refluxed for 24 h. After cooling, the reaction mixture was poured into water. The yellow solid was then recrystallised from methanol. Yield: 6.9 g (40%), mp 161°C. ^{1}H NMR (DMSO-d_6, ppm): $\delta =$ 2.05 (s; 3H), 7.78 (d; 2H), 8.05 (d; 2H), 8.23 (dd; 1H), 8.38 (d, 1H), 8.73 (d, 1H), 10.3 (s; 1H).

2-(4-aminophenyl)-5-nitrothiobenzimidazole 3: 11.5 g (0.037 mol) of 2 was added to a mixture of 120 ml of water, 25 ml of hydrochloric acid and 40 ml of ethanol and heated under reflux for 16 h. After cooling, the precipitated reaction product was filtered, washed with ether and dried. Yield: 5.75 g (57.5%), mp 240.3–241.2°C. ^{1}H NMR (DMSO-d_6, ppm): $\delta = 6.1$ (s; 2H), 6.65 (d; 2H), 7.75 (d; 2H), 8.15 (dd; 1H), 8.25 (d; 1H), 8.6 (s; 1H).

2-[4-[4-(N-methyl-N-(2-hydroxyethyl)amino)phenylazo]-phenyl]-4-nitrothiobenzimidazole e: Amine 3 was diazotised with sodium nitrite/hydrochloric acid and transformed into its tetrafluoroborate salt; 3.9 g (0.0108 mol) of this salt was reacted with 1.63 g (0.0108 mol) of *N*-methyl-*N*-(2-hydroxyethyl)aniline in 35 ml of *N*,*N*-dimethylformamide for 1 h at 0°C. The reaction mixture was then poured into iced water, filtered and washed out with ether, then dried. The reaction product was pure enough to be used in the next step (linking to the polymer backbone). Yield: 3 g (64%), mp 184.6–185.7°C. ^{1}H NMR (DMSO-d_6, ppm): $\delta = 2.85$ (t; 2H), 2.95 (t; 2H), 3.15 (s; 3H), 6.9 (s; 1H), 7.6 (d, 2H), 7.8 (d; 2H), 8.0 (d; 2H), 8.3 (m; 3H), 8.45 (d; 1H),8.85 (d, 1H).

2.5. Polymer synthesis

The precursor polymers were synthesised by copolymerisation of recrystallised maleimide and 4-phenylstyrene, indene, 1-adamantyl methacrylate respectively in *N*,*N*-dimethylformamide as solvent with 1 wt% of 2,2′-azobisisobutyronitrile at 65°C for 24 h under argon atmosphere, in 50:50 mol ratio. The copolymers were isolated by precipitation in methanol, collected by filtration, redissolved in DMF and reprecipitated in methanol, filtered and dried under vacuum.

2.6. Chromophore functionalisation of precursor polymers

General procedure: Diethylazodicarboxylate (DEAD) (20 mol% in excess towards mol of maleimide content to functionalise in the copolymer) is added dropwise to a cooled (0°C) solution of copolymer, triphenylphosphine (same content as DEAD) and hydroxyalkyl chromophore (a–e, equivalent content as mol maleimide to functionalise) in 1-methyl-2-pyrrolidinone for PM2 and PM3, THF for PM1 and a mixture of THF/1-methyl-2-pyrrolidinone for PM1e.

3. Results and discussion

The synthesis of chromophore functionalised maleimide copolymers PM1, 2 and 3 was a two step process. The prepolymers were obtained by radical polymerisation of maleimide with 4-phenylstyrene (PM1), indene (PM2) or

Table 1
Properties of chromophore functionalised PM1, 2 and 3

Polymer	wt% (max)[a]	$\bar{M}_n$ (10^4 g/mol)[b]	D[c]	T_g (°C)	T_d (°C)[d]
PM1b	38.1 (63.8)	3.8	2.2	224.5	> 320
PM1e	39.8 (60.1)	3.6	3.3	222	> 320
PM2a	17.1 (71.7)	1.3	1.9	287.5	> 350
PM2b	13.1 (71.7)	0.8	3.1	288	> 350
PM3a	35.6 (43.6)	2.2	2.7	239.5	> 320
PM3b	17.7 (43.6)	3.2	1.7	252	> 320
PM1c-1	39 (54.4)	2.1	3.0	224	> 350
PM1c-2	29 (54.4)	2.1	3.0	241	> 350
PM1c-3	17 (54.4)	2.0	2.9	254	> 350
PM1d-1	20 (62.2)	1.5	2.5	259	> 350
PM1d-2	18 (62.2)	1.8	2.7	261	> 350
PM1d-3	14 (62.2)	2.1	2.5	262	> 350

[a] Weight percent of NLO-dye in polymer from ^{1}H NMR and spectrophotometric measurements (and maximum weight percent).

[b] Apparent molecular weights measured by GPC in THF, polystyrene standards.

[c] Polydispersity: $D = \bar{M}_w/\bar{M}_n$.

[d] Decomposition temperatures.

Table 2
Wavelength of maximum absorption (λ_{max}) and second-harmonic coefficients d_{33} and $d_{33}(0)$

Polymer	λ_{max} (nm)[a]	$d_{33}(\omega)$ (pm/V)[b]	$d_{33}(0)$ (pm/V)[c]
PM1b	443	16	4.1
PM1e	452	11.4	2.6
PM2a	435	4.8	1.3
PM2b	446	6.3	1.5
PM3a	435	8.3	2.3
PM3b	446	5.8	1.4
PM1c-1	497	8.4	0.9
PM1d-2	496	2.7	0.3

[a] Measured in spincoated films.

[b] Measured at 1064 nm.

[c] Extrapolated to zero frequency using the frequency factor $\omega_{eg}^4/[(\omega_{eg}^2 - 4\omega^2)(\omega_{eg}^2 - \omega^2)]$ (obtained from the two level model, with ω_{eg} the frequency of the charge transfer band of the chromophore and ω the excitation frequency) [21].

adamantyl methacrylate (PM3) and transformed into their respective functionalised copolymers via a Mitsunobu reaction with hydroxyalkyl chromophores a–e.

Polymers PM1c and PM1d with a varied concentration of chromophore were obtained in this way. The copolymers are soluble in 1-methyl-2-pyrrolidinone, dimethylformamide and cyclohexanone.

The structure of the copolymers is given in Fig. 3.

As the maleimide is only partly functionalised, the real structure is a terpolymer with nonfunctionalised maleimide moieties in the copolymer structure.

The weight percent of NLO-dye in the copolymers, molecular weights $\bar{M}_n$, polydispersities (D), T_g's and decomposition temperatures (T_d) are given in Table 1. The prepolymers show an incorporation of the maleimide unit in poly(maleimide-indene) of 54.7 mol%, poly(maleimide-1-adamantyl methacrylate) of 29.3 mol%, while in poly(maleimide-4-phenylstyrene) 50 mol% is incorporated; for the latter we obtained alternating copolymers.

Very high glass transition temperatures for the nonfunctionalised copolymers could be measured: 269°C for PM1, 311°C for PM2 and 282°C for PM3.

The glass transition temperatures of the NLO-phore functionalised polymers are slightly lower, between 222 and 262°C, but nevertheless comparable to those of other NLO polymer materials reported in literature [4,9,12,13,18]. The rigidity of the main chain leads to high T_g's, which is a perquisite for useful and stable second-order NLO materials. Furthermore the decomposition temperatures are at least 70°C higher than the respective T_g's which prevents decomposition upon poling.

The highest T_g's are obtained for the non-functionalised as well as for the functionalised indene copolymers. They possess the most rigid main chain and their relatively low functionalisation degree favours higher T_g's.

The overall decrease in T_g of the functionalised copolymers compared to the nonfunctionalised ones is due to the interaction between the incorporated chromophore units; for example, the decrease in glass transition temperatures of PM1e and PM1d with increasing chromophore load is due to an increase in the interaction between the chromophore units and a decreased possibility of hydrogen bond formation.

The decrease in T_g of PM1d with the increasing functionalisation degree is not so well pronounced because the smaller spacer in the chromophore makes the system more rigid and the variation in NLO-load is rather small.

We must also notice that functionalisation of PM1 with chromophore d results in a maximum of about 30% of chromophore units which can be linked to the maleimide unit under Mitsunobu conditions. The reason is a possible secondary reaction of hydroxyalkyl chromophore d with DEAD which results in the formation of a hydrazine derivative [19]. This reaction was also stated by Mitsunobu. The degree of functionalisation however can be improved by the addition of neopentylalcohol to the reaction medium [20]. In this way the concentration of NLO-phore content could be increased to 60% of the maximum loading capacity.

An alternative method could have been the polymerisation of fully functionalised (NLO-phore c) maleimide monomer with 4-phenylstyrene or indene. However, this results in low molecular weight copolymers with high polydispersities and relatively low glass transition temperatures. For example, a molecular weight of 2000, polydispersity of 5.4 and T_g of 198°C was obtained for the 4-phenylstyrene copolymer and a molecular weight of 5300, polydispersity of 6.5 and T_g of 209°C was found for the indene copolymer. The maximum weight percent in chromophore load was obtained but no good quality films could be spincoated. Therefore the synthesis of prepolymers (copolymaleimides) seemed to us the most adequate method.

Eight from the 12 polymer systems could be spincoated

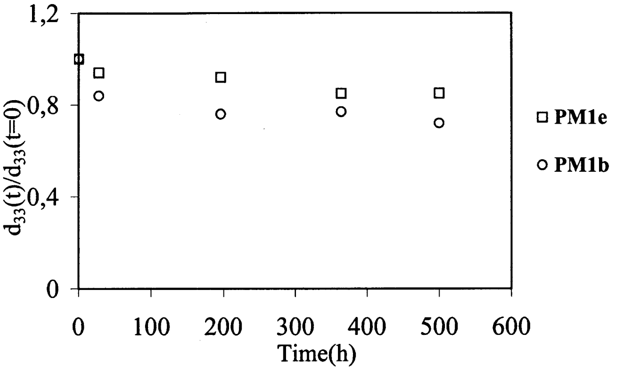


Fig. 4. Plot of the normalised second-harmonic coefficients as a function of time at 125°C of polymers PM1e and PM1b.

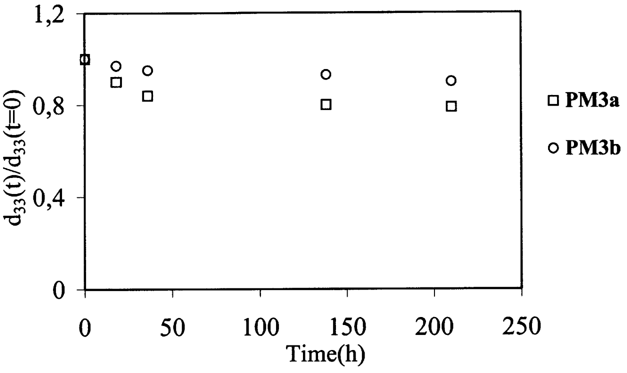


Fig. 6. Plot of the normalised second-harmonic coefficients as a function of time at 125°C of polymers PM3a and PM3b.

from cyclohexanone solution onto ITO glass substrates, yielding high quality films. The samples were heated under vacuum during several days to remove any residual solvent. The film thickness was measured with a DEKTAK 2 profilometer. Noncentrosymmetry in the polymer films was induced by corona poling at a temperature of 10°C below T_g. The SHG results are summarised in Table 2. The polymers have d_{33} values between 2.7 and 16 pm/V (measured at 1064 nm). Since the second harmonic wavelength was 532 nm, which is rather close to the absorption region of all chromophores, the values of $d_{33}(\omega)$ are resonantly enhanced and should be corrected for absorption. Using the two level model [21] we obtained nonresonant $d_{33}(0)$ values of 0.3–4.1 pm/V. In previous studies [12,13] on chromophore functionalised poly(maleimide-styrene)s, values of $d_{33}(0)$ even up to 23 pm/V were observed, while we measured now only moderate values. Several possible reasons for this phenomenon are: (a) in a typical corona-poling experiment, the effective poling field across the polymer film is unknown, (b) the high poling temperatures used increase the conductivity of the polymer samples and the thermal randomisation energy, resulting in a lower poling efficiency and second harmonic generation effect, (c) restricted mobility of the chromophores; this because of the rigid structure of the copolymers could be another reason and (d) most of the materials consist of moderate chromophore loading levels.

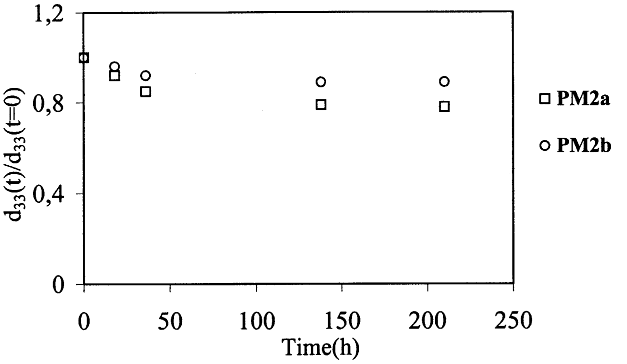


Fig. 5. Plot of the normalised second-harmonic coefficients as a function of time at 125°C of polymers PM2a and PM2b.

On the other hand the $d_{33}(\omega)$ values we obtained are higher or at least comparable with the values obtained by Sung et al. [22] for (*N*-azo-dye)maleimide-styrene copolymers, who observed d_{33} values of 3–5 pm/V (1064 nm). Chang et al. [23] observed values of 16.2 pm/V (1064 nm) for chromophore functionalised *N*-phenyl maleimide-methyl methacrylate, but they did not report the non resonant values. Günther et al. [24] obtained nonresonant d_{31} values of 23 pm/V (1064 nm) which corresponds to a value of d_{33} of ~60 pm/V if d_{33}/d_{31} should have been ≈3, however $d_{33}(0)$ values were not reported. High nonlinearity is not the only factor which is important for polymer materials in order to be useful in the field of electro-optics and photonics but the stability of the NLO effect in function of time at elevated temperatures is crucial for practical applications in optical devices.

Therefore one of the motivations to synthesise high T_g chromophore functionalised polymaleimides was the expectation that these materials would exhibit high stability at elevated temperatures.

A plot of $d_{33}(t)/d_{33}(t = 0)$ as a function of time, where $d_{33}(t)$ and $d_{33}(t = 0)$ represent the second-harmonic coefficient at times t and 0, respectively, versus time at 125°C is shown in Figs. 4–6. For all polymers, it can be seen that after a fast initial decrease, the second-harmonic efficiency does not significantly change over the time frame of the measurement. For polymers PM1b and 1e, there is 72 and 85%, respectively of remaining NLO activity after 500 h of heating, while for the other polymers we observe between 78 and 90% of the original NLO activity.

These polymers show extremely high stability for thermoplastic non-crosslinked systems, which may be compared with the results obtained in a previous study by us [12].

Compared to the stability measurements obtained by Sung et al. [22], who measured 20% of the remaining optical response after 18 h at 130°C and Günther et al. [24] who measured a decrease of 20% of the original SHG value after

10 min at 80°C, the polymaleimide systems we studied show much higher poling order stabilities. Our results show stabilities which may be compared with those obtained by Yu et al. [25] for polyimide systems which give 83% of remaining NLO response at 150°C.

Polymers PM1c-1 and PM1d-2, on the other hand, result in only 47% of remaining NLO-response. Although a much lower stability is observed than for the previous ones this result is still agreeable.

Hence, considering the acceptable second-order susceptibility values of the polymer systems investigated, combined with the good stability of the NLO response, we conclude that poly(maleimide-4-phenylstyrene)s, poly(maleimide-indene)s and poly(maleimide-adamantylmethacrylate)s are promising polymer materials for use in practical applications.

4. Conclusions

We synthesised chromophore functionalised polymaleimide copolymers by reaction of poly(maleimide-4-phenylstyrene), poly(maleimide-indene) and poly(maleimide-adamantyl methacrylate) prepolymers with hydroxyalkyl chromophores under Mitsunobu conditions. Second-order susceptibility values (d_{33}) up to 16 pm/V were obtained. The polymers exhibit high glass transition temperatures of 222–288°C which results in stable NLO-response at elevated temperatures. Four of the polymer systems lost only 10–22% of their nonlinearity after 210 h while PM1e and PM1b lost 15 and 28%, respectively, after 500 h at 125°C. In view of construction of devices these thermoplastics are promising materials.

Acknowledgements

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