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Isolation of poly(propylene glycol)s from water for quantitative analysis by reversed-phase liquid chromatography

Joanna Rychłowska, Agnieszka Zgoła, Tomasz Grześkowiak, Zenon Łukaszewski*

Institute of Chemistry, Poznań University of Technology, ul. Piotrowo 3, 60-965 Poznań, Poland

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Abstract

Procedures for the isolation of poly(propylene glycol)s (PPGs) from a water matrix have been developed. Solid-phase extraction with an octadecylsilica cartridge and elution with methanol or with a graphitised carbon black cartridge and elution with a mixture of dichloromethane–methanol (4:1) or liquid–liquid extraction with chloroform were all suitable for model samples. However, only liquid–liquid extraction was suitable both for model and real environmental samples. Methods for reversed-phase liquid chromatographic determination of PPGs based on derivatisation and ultraviolet or fluorescence detection have been developed. Four derivatisation agents [3,5-dinitrobenzoyl chloride, phenyl isocyanate, 1-naphthoyl chloride and 1-naphthyl isocyanate (NIC)] were tested. Only NIC was found to give good reproducibility as well as a satisfactory detection limit. Finally, a method with liquid–liquid extraction with chloroform, derivatisation with NIC and liquid chromatographic separation with fluorescence detection was established. The developed method shows a highly correlated linearity of the analytical signals of particular homologues within a wide concentration range (approximately from 0.01 to 10 mg l^{-1}). The precision of measurements is satisfactory for homologues having 5–9 oxypropylene subunits and becomes worse with an increase in the number of oxypropylene subunits. The limit of detection is 2 μg l^{-1} for the majority of homologues. The method is suitable for the isolation and quantitative determination of PPGs in river water samples and as a tool for biodegradation testing.

Keywords: Water analysis; Environmental analysis; Poly(propylene glycol); Surfactants

1. Introduction

Nonionic surfactants are the major group of surfactants. Among them, ethylene glycol derivatives are the most popular. Also propylene glycol derivatives have found their place in the commercial market because of their unique properties. Oxypropylene subunits are used mainly in the form of block copolymers with oxyethylene subunits. Three types of surfactants of this group are used:

$H–(OE)_x–(OP)_y–(OE)_x–OH$

$H–(OP)_y–(OE)_x–(OP)_y–OH$

$R–(OE)_x–(OP)_y–OH$

where R is an alkyl group, OE is an oxyethylene subunit ($–O–CH_2CH_2–$) and OP is an oxypropylene subunit ($–O–CH(CH_3)CH_2–$).

The oxypropylene block provides the molecule with its necessary hydrophobicity, while the oxyethylene block gives hydrophilicity. Different properties of the copolymers can be obtained by making changes in the ratio of oxypropylene to oxyethylene blocks. Surfactants of the OEOPOE type are better emulsifiers and dispersants [1–4], cover a broader range of molecular weights and are terminated by primary hydroxyl groups, giving higher reactivity and acidity. The surfactants of the OPOEOP type exhibit both lower foaming and reduced gelling tendencies, are better defoamers and are terminated by secondary hydroxyl groups giving lower reactivity and acidity [1].

In surfactants of the ROEOP type, the alkyl group provides the molecule with its necessary hydrophobicity. As a result a biodegradable product with low foaming and good wetting properties is obtained. The primary degradation of

* Corresponding author. Fax: +48-616652571.
E-mail address: zenon.lukaszewski@put.poznan.pl (Z. Łukaszewski).

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the fatty alcohol block copolymers is supposed to occur via a combination of two mechanisms, that of ω-hydrophile oxidation and also hydrophobe-hydrophile scission. As a result, poly(propylene glycol)s (PPGs) can be released to the environment. Although PPGs are non-toxic, their degradation is considered slow and leads to unknown products [5].

Polyglycols can be determined by numerous techniques. Among them, HPLC appears to be the most useful. Using this technique polyglycols can be determined in their native structure and after derivatisation of their hydroxy end-groups. The detection of underivatised polyglycols with the most popular UV absorbance detector is problematic due to lack of proper chromophores in their molecules. Analysis can be done only below 200 nm where baseline problems are inevitable and sensitivity is low. HPLC with refractometric detection [6–9] and evaporative light scattering detection [9–12] is an excellent tool for the determination of the molecular mass distribution or the composition of products. However, HPLC with these detection method was not used for the determination of PPG in environmental samples because of relatively low sensitivity. Liquid chromatography–mass spectrometry (LC–MS) [12–18] with different methods of soft ionisation (electrospray, thermospray) has great potential. This technique was successfully used for the identification of biodegradation intermediates of poly(ethylene glycol) (PEG) and ethoxylates.

Derivatisation of compounds with hydroxy end-groups with subsequent UV detection is a good alternative [10,19–23]. Derivatisation not only provides the possibility to use the UV detector but also improve the detection limit by allowing the possibility of fluorescence detection [19]. Among derivatisation agents, benzoyl chloride [17,19,23], 4-nitrobenzoyl chloride [19], 3,5-dinitrobenzoyl chloride (DNBC) [20,21], and phenyl isocyanate (PIC) [19,22] were frequently used to achieve UV detection of ethoxylated surfactants and polyglycols. Fluorescence detection of these compounds was achieved after derivatisation with 1-naphthyl isocyanate (NIC) [19] and 1-naphthoyl chloride (NC) [19,24]. However, only DNBC [10] was used for derivatisation of PPGs.

The presence of surfactants and their degradation products in environmental waters is of great concern. Most of the work is dedicated to the analysis of nonylphenol ethoxylates, fatty alcohol ethoxylates and their degradation products [13,14,16–22,24] which are often found in surface waters. Only a small number of papers have described PPG analysis [10,11,25]. None of them concerned quantitative analysis of PPGs in environmental samples. The aim of this work was to develop a suitable derivatisation procedure for the HPLC determination of PPGs as well as develop an efficient procedure for the isolation of PPGs from environmental samples (surface water, raw and treated sewage). The quantitative analysis of PPGs at the $\mu g\,l^{-1}$ level will also be presented.

2. Experimental

2.1. Reagents and chemicals

Poly(propylene glycol)s—PPG 425 from Fluka (Buchs, Switzerland), PPG 725 from Aldrich (Milwaukee, WI, USA), propylene glycol from U.S.P.C. (Rockville, MD, USA) and tripropylene glycol from Fluka—were used as received. DNBC, NC, PIC (all from Fluka) and NIC (from Aldrich) were used for derivatisation. HPLC-grade methanol from J.T. Baker (Deventer, The Netherlands) was used for HPLC measurements.

Chloroform and ethyl acetate all from POCh (Gliwice, Poland), dichloromethane from Fluka, acetonitrile and formic acid from J.T. Baker as well as sodium chloride and sodium hydrogencarbonate (both from POCh) of analytical grade were used.

The HPLC-grade water was prepared by reverse osmosis in a Demiwa system from Watek (Ledec nad Sazavou, Czech Republic), followed by double distillation from a quartz apparatus. Only freshly distilled water was used.

2.2. Derivatisation

2.2.1. 3,5-Dinitrobenzoyl chloride

To a sample containing 100 μg of PPG 425 in 200 μl of acetonitrile, 30 μl of a solution containing 10 mg of DNBC in 1 ml of acetonitrile and 1 μl of pyridine were added. The sample was heated for 50 min at 60 °C. After cooling the solution, 10 μl of methanol was added and the sample was heated again for 50 min at 60 °C to allow methanol to react with an excess of DNBC. The sample was evaporated and reconstituted to 1 ml of a mixture of methanol–water (4:6).

2.2.2. 1-Naphthoyl chloride

A 0.5 μl volume of NC and 0.5 μl of pyridine was added to 200 μl of acetonitrile containing 100 μg of PPG 425. The sample was heated for 1 h at 90 °C. After cooling the solution, 10 μl of methanol was added and the sample was heated again for 1 h at 90 °C. The resulting solution was evaporated and reconstituted in 1 ml of a mixture of methanol–water (4:6).

2.2.3. Phenyl isocyanate

To a sample containing 40 μg of PPG 425 in 200 μl of acetonitrile, 6 μl of PIC was added. The solution was heated for 30 min at 70 °C. Then 10 μl of methanol was added and the sample was heated again for 30 min at 70 °C. Finally, the solution was evaporated and the sample reconstituted in 1 ml of a mixture of methanol–water (4:6).

2.2.4. 1-Naphthyl isocyanate

A 6 μl volume of NIC was added to a sample containing 40 μg of PPG 425 in 200 μl of acetonitrile. The solution was sonificated for 20 min at 35 °C. The reaction was quenched by the addition of 10 μl of methanol with subsequent 10 min

Table 1
Gradient and detector conditions for poly(propylene glycol)s derivatised with four derivatisation agents

Derivatisation agent	Gradient	UV detection wavelength (nm)	Fluorescence excitation (Ex) and emission (Em) wavelength
3,5-Dinitrobenzoyl chloride	0 min 40% methanol 15 min 100% methanol	230	–
1-Naphthoyl chloride	0 min 50% methanol 10 min 100% methanol	220	Ex 220 Em 383
Phenyl isocyanate	0 min 40% methanol 15 min 100% methanol	235	–
1-Naphthyl isocyanate	0 min 60% methanol 23 min 100% methanol 24 min 100% methanol	222	Ex 225 Em 362

sonification at 35 °C. The sample was evaporated and reconstituted to 1 ml of a mixture of methanol–water (6:4).

2.3. Chromatography

A chromatographic system from Dionex (Sunnyvale, CA, USA) delivered by Polygen (Gliwice, Poland) consisting of a P580 A LPG gradient pump, ASI-100 autosampler, STH 585 oven, UV-Vis 170S detector and RF 2000 fluorescence detector was used. Twenty-five microliters samples were injected into a 250 mm × 4.6 mm i.d. analytical column packed with 5 μm Hypersil BDS C_{18} from ThermoQuest (Austin, TX, USA) delivered by Polygen with a guard column packed with 3 μm Hypersil BDS C_{18} from the same supplier. The column was flushed in a water:methanol gradient at a flow-rate of 1.5 ml min^{-1} at 35 °C. The gradients and detection conditions were different for all four types of PPG derivatives as described in Table 1.

2.4. Mass spectrometry

A 1100 chromatographic system from Agilent Technologies (Palo Alto, CA, USA) with an electrospray ionisation mass spectrometry (ESI-MS) detector was used. Fifty microliters samples were injected into a CC 250 mm × 4.0 mm i.d. 100-3 C_{18} Nucleosil analytical column from Macherey–Nagel (Düren, Germany). The mobile phase composition was 60% of 0.005 M formic acid in acetonitrile and 40% of 0.005 M formic acid in water. The flow-rate of the mobile phase was 0.7 ml min^{-1} at ambient temperature. The ESI-MS detector was operated in the positive-ion mode. The drying gas temperature was maintained at 350 °C at a flow of 13 l min^{-1}. The nebulisation pressure was 40 p.s.i.g. and capillary voltage 4000 V (1 p.s.i. = 68 g 4.76 pa). Full-scan MS chromatograms were obtained by scanning the quadrupole from 50 to 1000 *m/z* with the fragmentor set to 70 V.

2.5. Liquid–liquid extraction (LLE)

A 120 g amount of sodium chloride and 0.8 g of sodium hydrogencarbonate were added to a 400 ml water sample containing not more than 1 μg ml^{-1} of PPG. The sample was extracted with three portions of chloroform (40, 30 and 30 ml). A 10 ml aliquot of the combined extracts was evaporated to dryness and derivatised.

2.6. Solid-phase extraction (SPE)

Octadecylsilica (C_{18}) cartridges (Polar Plus C_{18}, 6 ml, 500 mg from J.T. Baker), polystyrene-divinylbenzene (PS-DVB) cartridges [Envi Chrom P, 6 ml, 250 mg from Supelco (Bellefonte, PA, USA)] and graphitised carbon black (GCB) cartridges–(Envi-Carb, 6 ml, 250 mg or 500 mg from Supelco) were used for SPE isolation. SPE cartridges were washed with 10 ml of methanol and conditioned with 7 ml of water. Without letting the cartridge dry, a 40 ml water sample containing 1 μg ml^{-1} of PPGs was applied at a speed of ca. 2 ml min^{-1}. The cartridge was dried and the sample eluted. Five milliliters of water miscible eluent or 1 ml of methanol followed by 4 ml of water immiscible eluent were used for elution of a sample.

2.7. Recommended procedure for PPG determination in environmental samples

A 120 g amount of sodium chloride and 0.8 g of sodium hydrogencarbonate were added to a 400 ml sample of filtered and unpreserved water containing not more than 1 μg ml^{-1} of PPG. The sample was extracted with three portions of chloroform (40, 30 and 30 ml). The chloroform extracts were collected in 100 ml volumetric flask and supplemented with chloroform to the mark. A 10 ml aliquot of the combined extracts was evaporated to dryness, reconstituted to 200 μl of acetonitrile and derivatised with NIC. Six microliters of NIC was added to a sample and the solution was sonificated for 20 min at 35 °C. The reaction was quenched by the addition of 10 μl of methanol with subsequent 10 min sonification at 35 °C. The sample was evaporated and reconstituted to 1 ml of a mixture of methanol–water (6:4). Twenty-five microliters were injected into the chromatographic column. The HPLC chromatogram was developed with a water–methanol gradient and the gradient and fluorimetric detection

conditions given in Table 1. Propylene glycol was used as a standard for quantification.

3. Results and discussion

3.1. Derivatisation

A successful analytical method requires efficient isolation of analytes from the water matrix as well as their quantification. The determination of PPGs by HPLC with UV absorption or fluorescence detectors strongly depends on successful derivatisation of PPGs due to the lack of chromophoric groups in their molecular structures. Therefore, several derivatisation agents were studied under different experimental conditions in order to achieve reproducible results. On the basis of the literature DNBC was initially used for derivatisation. However, both DNBC as well as the similar NC were found to be unsuitable because of poor reproducibility of results. PIC was then chosen for the derivatisation as a more reactive compound than both chlorides. Good reproducibility of the process was achieved. However, PIC was found unsuitable because of a huge peak of an unknown derivatisation product among PPG homologues peaks. NIC is less reactive than PIC and reproducibility of the derivatisation process could not be achieved using simple derivatisation. Fortunately, sonification of the mixture facilitated a good and fast derivatisation process. Moreover, the use of NIC enables the application of a fluorescence detector, which is more sensitive. The 1:2 mixture of PPGs 425 and 725 was derivatised with NIC and developed by HPLC with a gradient of the mobile phase. A good resolution of peaks was achieved, as shown in Fig. 1. A number of oxypropylene groups in all the homologues presented in Fig. 1 was deduced by coanalysis with tripropylene glycol. Propylene glycol was used as a standard for quantitative analysis.

3.2. Sample isolation

Isolation of PPGs with SPE cartridges and liquid–liquid extraction was investigated. PS-DVB, C_{18} and GCB cartridges were tested. Methanol, acetonitrile, ethyl acetate, chloroform and a mixture of dichloromethane–methanol (4:1) were used for the elution of adsorbed PPG homologues. In the case of the GCB cartridges, only dichloromethane–methanol (4:1) was used as recommended in the literature [10,26–30]. Chloroform was used as an organic phase in liquid–liquid extraction. Polydispersal PPG 425 containing homologues from 3 to 11 oxypropylene subunits was used as a test mixture.

Recoveries above 90% for all the homologues were achieved for liquid–liquid extraction with chloroform, for SPE with the C_{18} cartridge and elution with methanol as well as for SPE with GCB and elution with a mixture of dichloromethane–methanol (4:1). Results concerning SPE with the PS-DVB cartridge showed relatively good recoveries of approximately 85% with elution with the mixture of dichloromethane–methanol (4:1) (Table 2). Several additional observations are worth mentioning: (i) generally, higher homologues exhibit better recoveries than lower ones (see the C_{18} cartridge and elution with ethyl acetate or chloroform and the PS-DVB cartridge and elution with ethyl acetate or acetonitrile), (ii) the opposite effect and very low recoveries are observed with the C_{18} cartridge and elution with acetonitrile, (iii) in the case of the GCB cartridge, use of too large a cartridge (500 mg unit) leads to poorer recoveries. The developed method shows a highly correlated linearity of the analytical signals of particular homologues (from PPG 6 to PPG 18) within wide concentration range (approximately from 0.01 to 10 mg l^{-1}) (see Table 3). The precision of measurements is satisfactory for homologues having 5–9 oxypropylene subunits and becomes worse

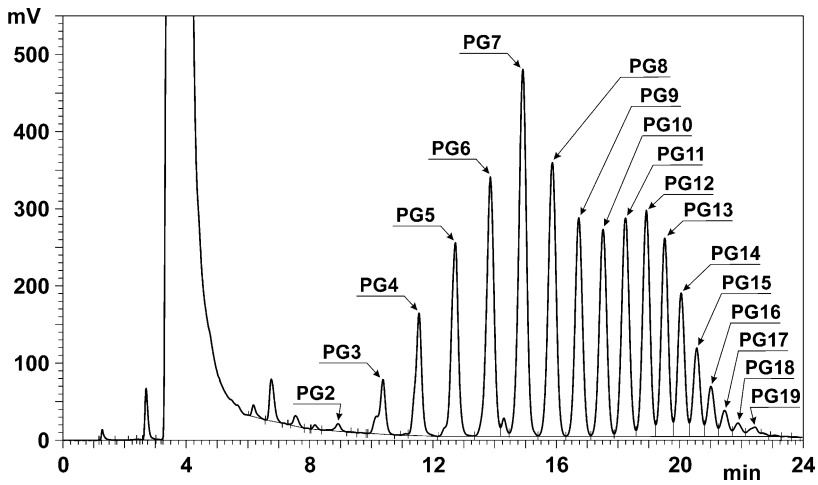

Fig. 1. A chromatogram of the 1:2 mixture of PPG 425 and PPG 725 derivatised with 1-naphthyl isocyanate obtained with a fluorescence detector according to conditions given in Table 1.

Table 2
Percentage recoveries of homologues of poly(propylene glycol)s 425 in liquid–liquid extraction (LLE) and solid-phase extraction (SPE) on octadecylsilica (C_{18}), polystyrene-divinylbenzene (PS-DVB) and graphitised carbon black (GCB)

Type of extraction	Solvents or mixtures used for elution or extraction	Recoveries of poly(propylene glycol)s with 4–11 oxypropylene groups ($n = 3$) (%)							
		PPG4	PPG5	PPG6	PPG7	PPG8	PPG9	PPG10	PPG11
LLE	Chloroform	90 ± 6	90 ± 7	89 ± 3	90 ± 3	89 ± 3	90 ± 4	89 ± 8	89 ± 11
SPE C_{18}	Methanol	94 ± 13	93 ± 4	92 ± 4	93 ± 6	94 ± 6	94 ± 7	95 ± 8	94 ± 10
	Acetonitrile	16 ± 1	9 ± 1	5 ± 1	4 ± 0.2	2 ± 0.4	2 ± 0.4	1 ± 0.3	1 ± 0.3
	Ethyl acetate	82 ± 3	84 ± 6	84 ± 4	89 ± 4	92 ± 5	95 ± 6	98 ± 7	97 ± 9
	Chloroform	82 ± 1	86 ± 3	91 ± 2	92 ± 2	95 ± 2	99 ± 2	100 ± 3	100 ± 4
	Dichloromethane–methanol (80:20)	78 ± 14	79 ± 14	80 ± 8	83 ± 13	83 ± 13	84 ± 14	86 ± 15	91 ± 17
SPE PS-DVB	Methanol	53 ± 7	52 ± 4	57 ± 4	56 ± 2	57 ± 4	56 ± 3	55 ± 5	58 ± 3
	Acetonitrile	78 ± 5	80 ± 4	83 ± 4	87 ± 5	90 ± 5	89 ± 7	92 ± 7	94 ± 12
	Ethyl acetate	77 ± 5	81 ± 5	87 ± 4	85 ± 3	85 ± 5	85 ± 7	89 ± 8	91 ± 9
	Chloroform	47 ± 16	50 ± 28	53 ± 28	56 ± 27	56 ± 28	55 ± 30	58 ± 32	67 ± 35
	Dichloromethane–methanol (80:20)	85 ± 6	87 ± 6	88 ± 5	88 ± 6	86 ± 8	84 ± 12	84 ± 14	81 ± 18
SPE GCB 500 mg	Dichloromethane–methanol (80:20)	90 ± 3	90 ± 4	87 ± 4	85 ± 5	84 ± 7	84 ± 8	82 ± 7	79 ± 8
SPE GCB 250 mg	Dichloromethane–methanol (80:20)	93 ± 2	93 ± 2	93 ± 2	93 ± 3	93 ± 3	94 ± 6	94 ± 8	93 ± 10

Table 3
Linearity, precision and limit of detection (LOD) for poly(propylene glycol) homologues having from 6 to 18 oxypropylene units

Homologue	Linearity range ($mg\,l^{-1}$)	Correlation coefficient	Precision (%)	LOD ($\mu g\,l^{-1}$)
PPG6	0.005–13	0.9997	3.1	1
PPG7	0.009–11	1.0000	3.3	2
PPG8	0.007–9	1.0000	3.7	2
PPG9	0.006–16	0.9997	4.5	2
PPG10	0.007–18	0.9997	8.4	2
PPG11	0.009–22	0.9997	9.7	2
PPG12	0.01–13	1.0000	11.4	2
PPG13	0.01–26	0.9997	13.5	2
PPG14	0.009–22	0.9997	15.9	2
PPG15	0.007–17	0.9997	18.8	3
PPG16	0.005–11	0.9997	21.5	3
PPG17	0.014–7	0.9995	25.0	7
PPG18	0.008–4	0.9995	26.7	10

with an increase in the number of oxypropylene subunits. The limit of detection (calculated on the basis of 3 S.D. of background) is $2\,\mu g\,l^{-1}$ for the majority of homologues.

3.3. Real sample analysis

After model studies were completed, both SPE and LLE were applied to real samples. Water samples from the River Warta (Poznań, Poland) were investigated, as well as treated sewage from biodegradation testing of PPG 725 under the conditions of the continuous flow activated sludge simulation test. SPE with the GCB cartridge and liquid–liquid extraction with chloroform were used. In order to check the recovery of PPG in real samples, a suspension of activated sludge was spiked with $40\,\mu g\,ml^{-1}$ of PPG 725, then filtered and the filtrate processed both by SPE with the GCB cartridge and liquid–liquid extraction. Recoveries of particular homologues of PPG are given in Table 4. Surprisingly, the results concerning the GCB cartridge were very poor. Similarly poor results were obtained with SPE with the C_{18} cartridge. However, the results of liquid–liquid extraction with chloroform were quite satisfactory. Thus, further determination of PPGs in real samples was performed with liquid–liquid extraction only. Co-adsorption of organic matter from real samples is probably the reason for the poor recoveries of PPG homologues in SPE isolation.

Table 4
Percentage recoveries of homologues of poly(propylene glycol) extracted from the filtrate of activated sludge suspension spiked with $40\,\mu g\,ml^{-1}$ of PPG 725

	PPG8	PPG9	PPG10	PPG11	PPG12	PPG13	PPG14	PPG15	PPG16	PPG17	PPG18	PPG19
Liquid–liquid extraction with chloroform	89.7	99.6	99.7	99.7	99.8	99.9	99.9	95.8	98.8	89.8	92.7	81.4
Solid-phase extraction on graphitised carbon black	10.8	15.5	14.2	13.1	11.5	9.9	8.1	6.5	5.1	4.7	5.2	6.0

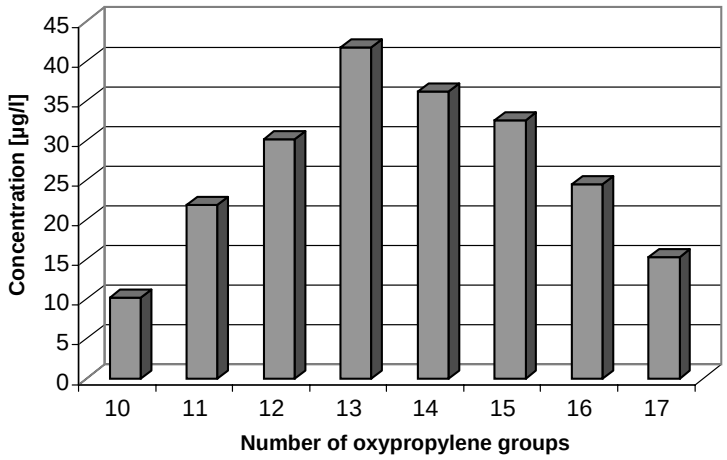

Fig. 2. A profile of poly(propylene glycol) homologue concentration in the River Warta, Poznań, Poland.

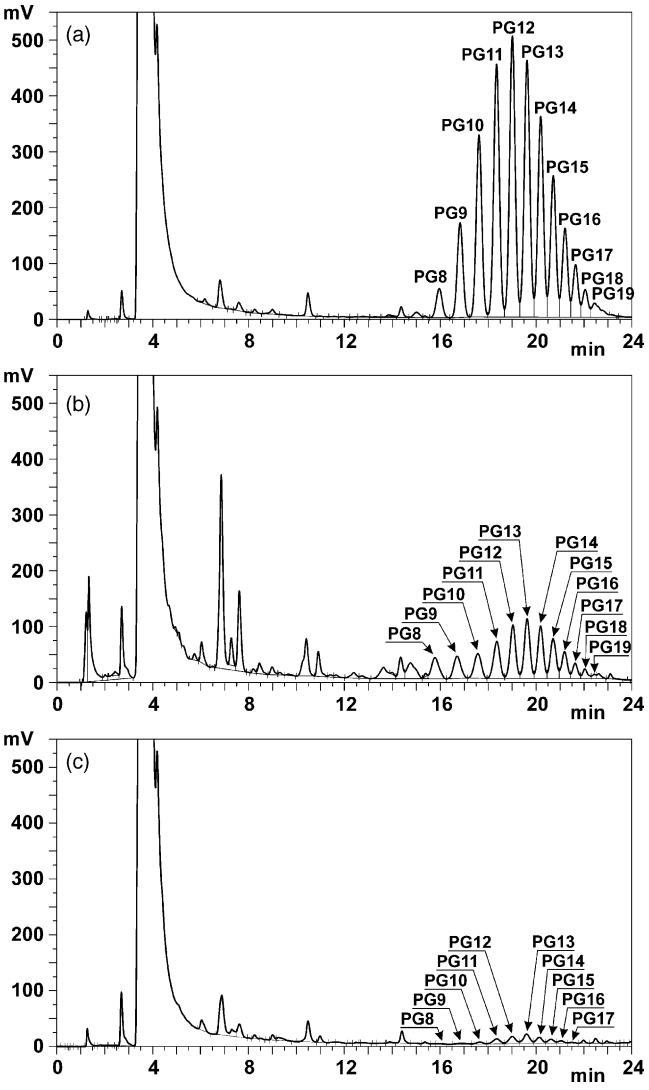

Fig. 3. Chromatograms of extracts of treated sewage in the continuous flow activated sludge simulation test of PPG 725: (a) starting point of test, (b) day 13 of test, (c) day 48 of test.

River water samples were extracted with chloroform and derivatised with NIC. The developed HPLC chromatogram clearly shows the presence of PPG homologues from 10 to 17 oxypropylene subunits with an overall concentration of 212 $\mu g\,l^{-1}$. The profile of PPG homologue concentrations in the River Warta is shown in Fig. 2. Concentrations were calculated using propylene glycol as an external standard. The possible error of such a determination may be removed when PPG homologues will be available as individual substances. Peak identification in fluorescence detection was made by spiking the sample with tripropylene glycol and PPG 425, as mentioned above. The presence of homologues containing 11 or more oxypropylene groups in chloroform extracts from river water samples was confirmed by mass spectrometry measurements. The characteristic series of equally spaced ions at *m*/*z* 616, 674, 732, 790, 848 and 906 ($[M + NH_4]^+$ type ions) was found.

3.4. Biodegradation test

The developed method was also used for the determination of PPG homologues in treated sewage from biodegradation studies of PPG 725, under the conditions of the continuous flow activated sludge simulation test. HPLC chromatograms of treated sewage from days 13 and 48 of the test, as well as at the beginning of the test are shown in Fig. 3. Progress in biodegradation of PPG 725 is clearly shown.

4. Conclusions

(1) Procedures for isolation of poly(propylene glycol)s from the water matrix have been developed. Solid-phase extraction with a C_{18} cartridge and elution with methanol or with a GCB cartridge and elution with a mixture of dichloromethane–methanol (4:1) is suitable for model samples but unsuitable in the case of real samples. Liquid–liquid extraction is suitable both for model as

well as environmental samples and is recommended for further use.

(2) A method for RP-HPLC determination of poly(propylene glycol)s based on derivatisation with 1-naphthyl isocyanate and fluorescence detection has been developed and successfully applied. The method is suitable for the quantitative determination of poly(propylene glycol)s in samples from river water and biodegradation tests.

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