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Evaluation of a new hypercrosslinked polymer as a sorbent for solid-phase extraction of polar compounds

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Abstract

A new hypercrosslinked polymer (HXLGp) with hydrophilic character due to the presence of hydroxyl moieties has been tested as a sorbent for the solid-phase extraction (SPE) of several polar compounds from water samples. This new sorbent enables the on-line extraction of 300 ml of sample with recoveries higher than 80% for polar compounds such as oxamyl, methomyl or desisopropylatrazine (DIA). The HXLGp has also been compared to other commercially available sorbents such as Oasis® HLB (hydrophilic macroporous), to hydrophobic hypercrosslinked resins and to a previously synthesized sorbent based on *N*-vinylimidazole-divinylbenzene. The results are consistently better with the new synthesized sorbent. The method was successfully applied to the on-line SPE-HPLC of tap and river water samples. The validation with river water samples provided good linearity range and detection limits between 0.03 for methomyl and 4-nitrophenol (4NP) to 0.2 $\mu g\,l^{-1}$ for phenol (Ph).

Keywords: Hydrophilic hypercrosslinked sorbent; Solid-phase extraction; Polar pollutants; Water analysis

1. Introduction

Relative to other available extraction methods, solid-phase extraction (SPE) is becoming more popular not only because of its ready adaptation to high throughput automation, but also because the increased availability of high quality SPE sorbents during the last few decades [1–4].

Various sorbents have been developed to extend the scope of the method and to facilitate the convenient processing of different sample types [1,3]. Thus, to increase the retention of polar compounds using polymeric sorbents, two influential parameters, specific surface area and hydrophilicity, can be conveniently tuned [5,6].

To take advantage of the retention through the specific surface area of the sorbents, macroporous copolymers (based on styrene-divinylbenzene copolymers), with surface areas up to $800\,m^2\,g^{-1}$, have been gradually replaced by the hypercrosslinked sorbents [7,8]. These hypercrosslinked materials firstly introduced by Davankov and Tsyurupa [9] posses both micropores and macropores, and display an exceptional specific surface area (over $1000\,m^2\,g^{-1}$) which intensifies the π–π interactions between the sorbent and the analyte [7,10–13]. Some commercially available hypercrosslinked polystyrene sorbents like Purosep 200 (Purolite), Chromabond® HR-P (Macherey-Nagel), Hysphere SH (Spark Holland), Isolute® ENV+ (IST) or Lichrolut® EN (Merck) have been tested in SPE and the recovery levels proved to be acceptable when moderately polar compounds were analyzed. However, the recoveries were low for the analysis of the most polar compounds, such as phenol (Ph) [14] or

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aniline [15]. On the other hand, in an early study [16] Tsyurupa et al. obtained good results in the extraction of phenol with some hypercrosslinked resins; however, in this study neither the method nor the concentration of phenol was pertinent to the analysis of e.g. environmental samples as it was in the previous studies [14,15].

The hydrophilicity of the sorbent is another determining factor, and thus can be adjusted by slight modification with polar functional groups [17,18] or by introducing a polar monomer into the polymer skeleton [5,6,19–21].

In previous work [6,20,21], we synthesized hydrophilic polymeric sorbents as conventional macroporous polymers using polar monomer (4-vinylpyridine, *N*-vinylimidazole or 4-vinylimidazole) and with specific surface areas up to 700 $m^2 g^{-1}$. The retention properties were found to increase with the degree of hydrophilicity, but also with the specific surface area. However, when large volumes of the analyte solution were passed through the sorbent, the small and polar compounds were not retained, suggesting that the morphology of the polymers should be improved.

Our next goal therefore was to improve the morphology of the polymer while maintaining the hydrophilicity and we synthesized [22] a hypercrosslinked polymer which contains some hydroxyl moieties in the skeleton, at the expense of a reduced specific surface area (908 $m^2 g^{-1}$) relative to that of the earlier hypercrosslinked resins.

In the present study we have evaluated in detail the retention behaviour of this new sorbent (HXLGp) for the SPE of a number of important polar compounds. Resin HXLGp was also compared to other sorbents. The performance of the method was also tested with real water samples.

2. Experimental

2.1. Reagents and standards

The pollutants selected to check the sorbent were classified in two groups. Group A is made up of oxamyl, methomyl, desisopropylatrazine (DIA), desethylatrazine (DEA), phenol, 4-nitrophenol (4NP) and (4-chloro-2-methyl-phenoxy) acetic acid (MCPA). Group B is made up of exclusively highly polar phenolic compounds, such as hydroquinone, resorcinol, cathecol, orcinol and guaiacol. The phenolic compounds were obtained from Aldrich (Steinheim, Germany) and the rest of compounds were supplied by Riedel-de-Haën (Seelze, Germany).

Standard solutions of 2000 mg l^{-1} of each compound were prepared in methanol. The mixture of all the compounds was prepared by diluting the standard solution with Milli-Q water (Millipore, Bedford, MA, USA).

HPLC-grade acetonitrile (ACN) (SDS, Peypin, France) and Milli-Q water were used to prepare the mobile phase. Hydrochloric acid was used to adjust the pH of the mobile phase and the sample, and sodium sulfite was added to decrease the matrix influence. Both were supplied by Probus (Badalona, Spain).

The reagents used in the polymerization—*para*-vinylbenzyl chloride (*p*VBC) (95%) was supplied by Dow Chemical (Middx, UK) and divinylbenzene (DVB) (80%) were from Aldrich (Steinheim, Germany) and 2,2′-azobisisobutyronitrile (AIBN) was from BDH (Poole, UK). Mowiol® (PVA) 23/88 (Mw ∼127.000, 88% hydrolyzed), ferric chloride (97%) and 1,2-dichloroethane (DCE) anhydrous (99.8%), were all from Aldrich (Steinheim, Germany) and sodium chloride from Fluka (Buchs, Switzerland).

2.2. Chromatographic equipment

The chromatographic experiments were performed with two LC-10AD_{VP} pumps, an on-line connected degasser DGU-14A and CTO-6AS column oven (all from Shimadzu (Tokyo, Japan)), an injection valve with a 20 μl loop and a Hewlett Packard (Avondale, PA, USA) Series 1100 UV spectrophotometric detector. The analytical column was a 250 mm × 4.6 mm i.d. stainless-steel column packed with Kromasil 100 C_{18}, 5 μm (Teknokroma, Barcelona, Spain).

The on-line solid-phase extraction system, which was connected to the chromatographic system by means of a six-port switching valve (Rheodyne, Cotati, CA, USA), consisted of a LC-10AS pump (Shimadzu, Tokyo, Japan) used to preconcentrate samples through a stainless-steel precolumn of 10 mm × 3 mm i.d. purchased from Free University (Amsterdam, The Netherlands). This was laboratory-packed with ∼40 mg of the 50–75 μm studied sorbent and was used for the on-line trace enrichment process.

2.3. Hypercrosslinking reaction

The 2 wt%DVB–98 wt%*p*VBC gel-type (2.5 g) precursor resin (obtained by the usual suspension polymerization method) and DCE (40 ml) were placed in a water-jacked round bottomed flask (100 ml), and the solution was left under nitrogen for 1 h to swell the beads. Then the ferric chloride (in a molar ratio 1:1 of CH_2Cl: $FeCl_3$) mixed in DCE (40 ml) was added to the solution. The final solution was then rapidly heated to 80 °C and kept at this temperature for 18 h [22].

The beads were then washed with water and methanol on a sieve (75 μm), and the resin was placed in a Soxhlet apparatus and extracted with acetone overnight. Finally the HXLGp resin was washed with methanol and diethyl ether in a filter funnel and dried in a vacuum oven for a day at 40 °C.

The resin was characterized by measuring its surface area (908 $m^2 g^{-1}$, N_2 sorption, BET) and the chlorine and oxygen content (2.13 wt.% Cl and 3.96 wt.% O) with elemental microanalysis. The oxygen content of the resin was characterized and attributed as in hydroxyl form due to the hydrolysis process during the synthesis of the precursor resin (2 wt%DVB–98 wt%*p*VBC gel-type) [22].

2.4. Chromatographic conditions

The mobile phase consisted of Milli-Q water acidified to pH 3.0 with hydrochloric acid and acetonitrile. The flow-rate was 1 ml min^{-1} and the temperature of the column oven was set at 65 °C. Each group of compounds was separated using different elution gradients. The gradient profile for group A was from 15% to 20% ACN in 10 min, 50% ACN in 10 min and 100% solvent in 15 min (held for 2 min), after which the mobile phase was returned to the initial conditions in 3 min. The gradient profile for group B was from 15% to 25% ACN in 10 min and 100% solvent in 5 min (held for 2 min), after which the mobile phase was returned to the initial conditions in 3 min.

The wavelengths used to detect the compounds were at 240 nm (oxamyl, methomyl, DIA, DEA and 4NP), at 280 nm (all the phenolic compounds studied, except 4NP) and at 230 nm (MCPA).

2.5. On-line solid-phase extraction

The in-house synthesized hypercrosslinked sorbent (HXLGp) and the commercial Oasis® HLB were laboratory packed (∼40 mg) in a 10 mm × 3 mm i.d. stainless steel precolumn used for the on-line trace enrichment in the solid-phase extraction process.

A Shimadzu LC-10AS pump with a switching valve was used to load the different volumes of both the solvent and the sample to be extracted. The protocol used, which was the same for the two sorbents (HXLGp and Oasis® HLB) and both groups of compounds, was the following: the SPE precolumn was conditioned by flushing with 6 ml of acetonitrile and 2 ml of acidified Milli-Q water (pH 3.0) at 3 ml min^{-1}; different volumes (10–300 ml) of the sample acidified with hydrochloric acid at pH 3.0 at 3 ml min^{-1} were extracted; the analytes trapped on the precolumn were desorbed in the backflush mode using only the organic solvent of the mobile phase (acetonitrile) instead of the mobile phase in the initial conditions [23].

Samples from the Ebre river and tap water were filtered through 0.45 μm nylon membranes (Supelco Inc. Bellefone, PA, USA) before the preconcentration step to eliminate the particulate matter. The optimum addition (500 and 1000 μl per 100 ml of tap and river water, respectively) of Na_2SO_3 (10%, w/v) was added prior to the preconcentration process in order to decrease the initial band caused by humic and fulvic acids in the real water samples [24].

3. Results and discussion

In this study we evaluate a new hydrophilic hypercrosslinked resin, which was previously synthesized and characterized by our group [22]. The preliminary assessment of this resin suggested that it would act as a very good SPE sorbent to extract polar compounds.

3.1. On-line trace enrichment

The recoveries in the on-line SPE process were first assessed for analytes in group A. Different sample volumes (10–300 ml) in Milli-Q water acidified at pH 3 (with HCl) were percolated through the precolumn packed with sorbent. These samples were spiked with the analytes of interest at different concentration, so that the amount injected was kept constant (0.2 μg) for each analyte.

The commercial Oasis® HLB sorbent, which is a macroporous copolymer made from a balanced ratio of two monomers, hydrophilic *N*-vinylpyrrolidone and lipophilic divinylbenzene, and possessing specific surface area of ∼800 m^2 g^{-1}, was chosen for comparison.

Table 1 shows the recoveries obtained with both sorbents. From the results we can observe no decrease in the recoveries for all the percolated volumes for the HXLGp sorbent, thus, recoveries higher than 80% (except for phenol for which recovery was 72%) were provided, even, when 300 ml of sample spiked with 0.6 μg l^{-1} were percolated.

The Oasis® HLB recoveries (Table 1) under the same conditions as HXLGp and for the largest volumes (i.e. 200 and 300 ml) were lower, especially for the most polar compounds of the group (i.e. oxamyl, methomyl, DIA and phenol). The recoveries with the HXLGp sorbent can be also compared positively under the same conditions to the results obtained with a previously synthesized sorbent based on *N*-vinylimidazole-divinylbenzene (NVIm-DVB) [21], which, similarly to Oasis® HLB, is a hydrophilic (6.3% wt. N) macroporous resin with an specific surface area of 627 m^2 g^{-1}. The recoveries provided by the NVIm-DVB, when 200 ml were percolated on-line under the same conditions as HXLGp, for oxamyl and methomyl were 55% and 37%, respectively, whereas for HXLGp the recoveries were 89% and 94%, respectively.

In view of the satisfactory results of the group A analytes with HXLGp, we tested the resin and also compared it to Oasis® HLB in the SPE of a group of highly polar compounds (group B). To do so, the on-line SPE was carried out as before,

Table 1
Recoveries obtained with the synthesized HXLGp sorbent and commercial Oasis® HLB in on-line SPE for different sample volumes spiked with the group A analyte mixture in Milli-Q water

Compound	Recovery (%)					
	HXLGp synthesized sorbent			Oasis® HLB		
	100 ml	200 ml	300 ml	100 ml	200 ml	300 ml
Oxamyl	89	89	86	80	62	53
Methomyl	90	94	90	75	46	41
DIA	87	85	84	83	69	63
Ph	88	80	72	72	60	52
DEA	88	89	88	83	96	91
4NP	85	97	85	97	91	90
MCPA	77	80	81	88	83	86

For all the conditions see text. % relative standard deviations (RSD) ($n = 3$) were lower than 7.

Table 2
Recoveries obtained with the synthesized HXLGp sorbent and commercial Oasis® HLB in on-line SPE for different sample volumes spiked with the group B analyte mixture in Milli-Q water

Compound	Recovery (%)							
	HXLGp synthesized sorbent				Oasis® HLB			
	10 ml	25 ml	50 ml	100 ml	10 ml	25 ml	50 ml	100 ml
Hydroquinone	32	29	26	13	40	20	13	7
Resorcinol	78	67	43	28	56	41	22	13
Catechol	36	19	15	9	68	59	36	23
Orcinol	87	80	78	72	75	71	57	39
Guaiacol	91	94	90	93	91	89	88	84

For all the conditions see text. % relative standard deviations (RSD) ($n = 3$) were lower than 15.

but with the group B analytes the percolated volumes were just up to 100 ml. As shown in Table 2, both sorbents display low recoveries for the most polar compounds, but the lowest values corresponded to those found using Oasis® HLB, except in the case of catechol. In spite of the low values obtained for HXLGp with the most polar compounds of the group B, the results can be considered as satisfactory. This is because we must bear in mind, for instance, that hydroquinone could not be extracted at all with any other commercial hypercrosslinked sorbent based on styrene-divinylbenzene, such as Amberchrom™ GC-161 m (900 $m^2\,g^{-1}$), Envichrom P (800–950 $m^2\,g^{-1}$) or Lichrolut® EN (1200 $m^2\,g^{-1}$) [25] when 100 ml of sample spiked with 2 $\mu g\,l^{-1}$ of the above analyte was on-line preconcentrated under the same conditions as ours. In addition, resorcinol, also tested with the above commercial sorbents, had recoveries lower than 16% (corresponding to Lichrolut® EN) [25]. Another good comparison is with hypercrosslinked resin (Amberchrom™ GC-161 m) chemically modified with 2-carboxy-3/4-nitrobenzoyl and 2,4-carboxybenzoyl moieties, and evaluated for their extraction behaviour under the same conditions for this group B of analytes [26]. The results after percolating 20 ml of sample with both chemically modified sorbents were for hydroquinone 10% and 11%, respectively; and for resorcinol, 23% and 22%, respectively. The new HXLGp sorbent therefore performs measurably better than these two modified hypercrosslinked resins.

Interestingly although Oasis® HLB and NVIm-DVB sorbents are hydrophilic materials with comparable specific surface area to that of HXLGp (800–627 $m^2\,g^{-1}$ respectively versus 908 $m^2\,g^{-1}$), HXLGp shows better recovery of the polar analytes studied here. These two resins are conventional macroporous types, and so this suggests that it is the specific microporous structure [27–29] of the hypercrosslinked HXLGp sorbent that is responsible for the enhanced recoveries rather than the hydrophilic nature and surface area. Why then should the two chemically modified hypercrosslinked resins also show poorer recoveries than HXLGp? All three of these sorbents are microporous and polar with similar surface area, and so in this case the difference may be due to a lower level of polarity in the modified resins [26], or due to the different specific chemical structures that give rise to the polarity. Finally in the case of comparison of the performance of HXLGp with that of the hydrophobic hypercrosslinked sorbent, Lichrolut® EN (surface area, 1200 $m^2\,g^{-1}$), it seems that the hydrophilicity of HXLGp gives rise to the better recoveries, and that the microporous structural effect is less important in this comparison.

3.2. Analysis of real samples

The method was applied to the on-line preconcentration of tap and river water. Before the analysis, 500 μl (for tap water) and 1000 μl (for river water) of 10% Na_2SO_3 solution for each 100 ml of sample [24] were added to decrease the initial band due to the humic and fulvic acids.

In testing the performance of the method with real samples we first examined compounds in group B, since these highly polar species have hardly ever been studied, and the results in Milli-Q water with our new synthesized hypercrosslinked sorbent were quite promising.

The recovery values (hydroquinone 11%; resorcinol 32%; catechol 11%; orcinol 63%; guaiacol 68%) obtained by percolating 50 ml of tap water sample spiked with the mixture of analytes at 2 $\mu g\,l^{-1}$ were quite similar to those obtained with Milli-Q water. However, as the chromatogram in Fig. 1 shows

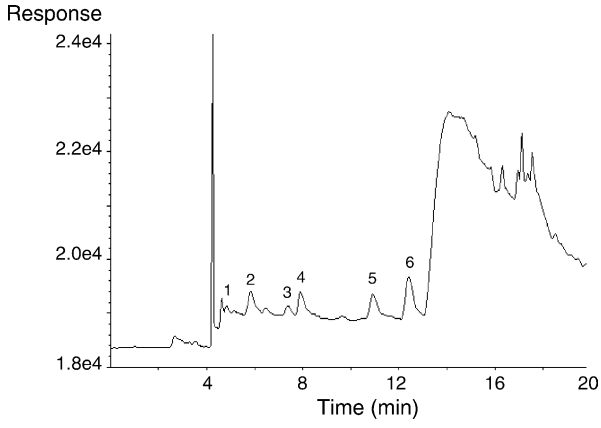

Fig. 1. Chromatogram obtained following on-line trace enrichment of 50 ml of tap water spiked at 2 $\mu g\,l^{-1}$ of the group B compounds and with the addition of 500 μl of 10% Na_2SO_3 solution for each 100 ml of sample. Peak designation: (1) hydroquione, (2) resorcinol, (3) catechol, (4) orcinol, (5) phenol and (6) guaiacol.

after percolating 50 ml of tap water spiked with 2 μg l^{-1} of a standard solution of the group B analytes and phenol (which was used as control) there is peak broadening, which can be attributed to the high retention of these compounds. Hence, these peaks could not be properly integrated at lower concentrations. One option to overcome this peak broadening would be to perform the extraction of these compounds in the off-line mode. However, earlier studies [21] showed that volatile compounds such as phenol can be lost during the evaporation step, which has to be included after the off-line approach, to be able to analyse for such low levels of analyte as is possible in the on-line mode. Therefore, no further investigation involving this group of compounds was carried out.

In order to better assess the application of the method with the HXLGp sorbent, we then chose the group A analytes spiked in a sample volume of 300 ml, because of the satisfactory results achieved using this volume with Milli-Q water.

Fig. 2 shows the chromatograms for 300 ml of river water sample without (a) and with (b) the addition of 0.3 μg l^{-1} of the group A compounds and with the addition of 1000 μl per 100 ml of sample. As we can see, despite the use of the sulfite addition to reduce the initial band due to humic and fulvic acids, there still remains significant interference. This might be attributed to the large on-line preconcentrated volume (300 ml) of sample with a high charge of organic matter, such as in river water. Thus, when a 300 ml sample of tap water (less organic matter) was percolated (Fig. 3) without (a) and with (b) the addition of 0.3 μg l^{-1} the group A compounds and with the addition of 500 μl per 100 ml of sample,

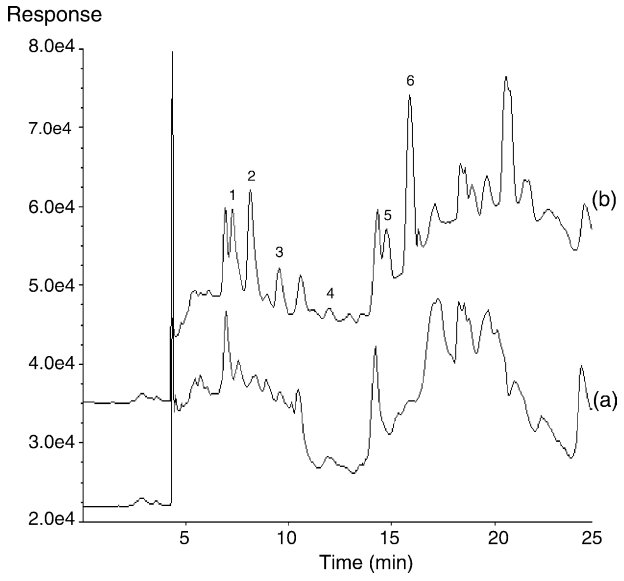

Fig. 2. Chromatograms obtained following on-line trace enrichment of 300 ml of Ebre river water without (a) and with (b) the addition of 0.3 μg l^{-1} of the group A compounds and with the addition of 1000 μl of 10% Na_2SO_3 solution for each 100 ml of sample. Peak designation: (1) oxamyl, (2) methomyl, (3) DIA (4) phenol, (5) DEA and (6) 4NP.

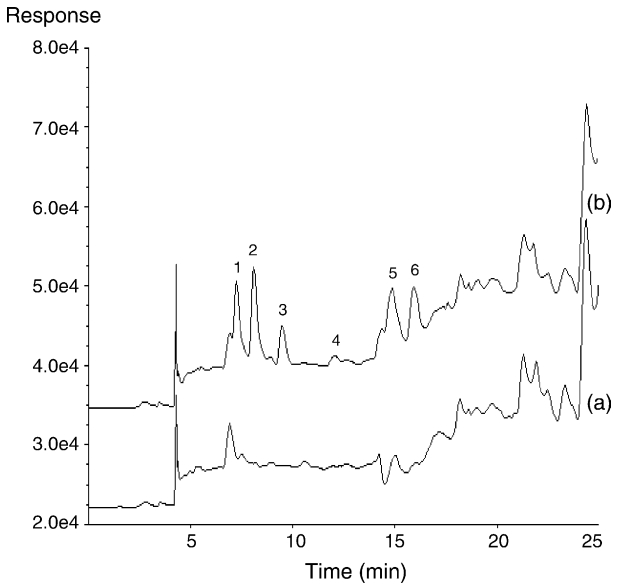

Fig. 3. Chromatograms obtained following on-line trace enrichment of 300 ml of tap water without (a) and with (b) the addition of 0.3 μg l^{-1} of the group A compounds and with the addition of 500 μl of 10% Na_2SO_3 solution for each 100 ml of sample. For peak designation, see Fig. 2.

less interference with a flatter baseline is seen in the chromatograms.

The recoveries obtained with river and tap water are summarized in Table 3. For all the analytes with real water samples these recoveries suffer a slight decrease compared to the recoveries with Milli-Q water and this is characteristic for the extraction of real samples, and this is most notable in the decrease in DIA's recovery. The decrease in the case of 4NP in tap water and DEA in river water can be explained by some interference present at the same retention time, which makes it difficult to integrate properly the response of these compounds. Another aspect related to the interferences is that MCPA could not be identified in the real sample, possibly because some interference masks this, and so the MCPA recoveries have not been included in Table 3.

The linearity of the response, detection limits (LODs) (calculated as the response for which the signal-to-noise ratio was

Table 3
Recoveries and RSD (n = 5) of the on-line SPE with the HXLGp sorbent for 300 ml of standard solution spiked with 0.3 μg l^{-1} of each compound in tap and Ebre river water

Compound	Tap water[a]		River water[b]	
	Recovery	RSD	Recovery	RSD
Oxamyl	79	4	84	3
Methomyl	69	3	75	2
DIA	55	5	55	1
Ph	70	10	66	12
DEA	87	6	66	8
4NP	68	6	86	7

All values in %.
[a] With the addition of 500 μl 10% Na_2SO_3 solution per 100 ml of sample.
[b] With the addition of 1000 μl 10% Na_2SO_3 solution per 100 ml of sample.

Table 4
Linear range and detection limits with on-line trace enrichment of 300 ml of Ebre river water at pH 3.0 and addition of 1000 μl 10% Na_2SO_3 for each 100 ml of sample

Compound	Linear range ($\mu g\,l^{-1}$)	r^2	Detection limit ($\mu g\,l^{-1}$)
Oxamyl	0.30–6.0	0.9991	0.1
Methomyl	0.06–8.0	0.9982	0.03
DIA	0.10–6.0	0.9985	0.06
Ph	0.30–8.0	0.9982	0.2
DEA	0.20–4.5	0.9975	0.1
4NP	0.06–8.0	0.9987	0.03

3), repeatability and reproducibility (between days) for the total analytical system, including the preconcentration step with the HXLGp sorbent, was checked with 300 ml of spiked Ebre river water with the addition of 1000 μl of 10% Na_2SO_3. Table 4 shows the linear range, correlation coefficients and LODs. The method's repeatability and reproducibility, expressed as the relative standard deviation (RDS) of five analyses of 300 ml of Ebre river water spiked at 0.3 $\mu g\,l^{-1}$ were lower than 12% for all the compounds examined.

4. Conclusions

This study demonstrates that the surface polarity and the morphology of the new hypercrosslinked resin sorbent HXLGp are both important in controlling the retention of polar compounds. In practice, the recoveries of polar compounds are higher than when using hydrophilic conventional macroporous or hydrophobic hypercrosslinked sorbents.

Polar compounds are efficiently extracted from 300 ml tap and river water samples after the optimum sulfite addition by on-line SPE with HXLGp, and quantitative recoveries were obtained.

The method was validated for 300 ml of Ebre river water spiked with the analyte mixture; and the linear range, detection limits, repeatability and reproducibility were satisfactory.

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