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Determination of polychlorinated biphenyl and polycyclic aromatic hydrocarbon marine regional Sediment Quality Guidelines within the European Water Framework Directive

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Marine regional Sediment Quality Guidelines (SQG) for polychlorinated biphenyls (PCB) and polycyclic aromatic hydrocarbons (PAH) from the Basque coast (SE Bay of Biscay) were determined, on the basis of sediment chemistry, toxicity and benthic community disturbance from analysis of 756 estuarine and coastal samples. The SQG were calculated using a percentile approach (based upon effect and non-effect data), using non-normalised and normalised concentration, by total organic carbon. However, normalisation by total organic carbon did not result in any improvement in accuracy compared with non-normalised values. Hence, this study proposes non-normalised SQG values for assessment of the chemical and physicochemical status in marine waters in the Basque Country, within the Water Framework Directive: 24.6–29 $\mu g\,kg^{-1}$ for total PCBs; 164–285 $\mu g\,kg^{-1}$ for low molecular weight PAH; 922–1537 $\mu g\,kg^{-1}$ for high molecular weight PAH; and 1607–2617 $\mu g\,kg^{-1}$ for total PAH.

Keywords: Sediment Quality Guidelines; PAH; PCB; Basque Coast; Water Framework Directive; marine sediments

1. Introduction

Two levels are adopted within the use of chemical indicators in the Water Framework Directive (WFD) [1]: (1) achievement of chemical status, i.e. being 'good', when priority substance concentrations lie below the Environment Quality Standards (EQS); and (2) classification of the physicochemical status of sediments, i.e. 'high' when the main pollutant concentrations are below background levels, 'good' when they range between the background level and EQS, and 'moderate' when they exceed EQS. The new Directive 2013/39/EU establishes EQS for priority substances for water and biota only. According to these Directives, European Union Member States should monitor these priority substances and main pollutants in sediments; they should be able to establish EQS at the national level (Article 3).[2] Deriving EQS for marine sediments is a difficult task.[2–4] Hence, several investigations have been undertaken to determine alternative approaches (i.e. Sediment Quality Guidelines, SQG [5–8] and Environmental Assessment Criteria[9]). In this way, SQG can be applied, not as a criterion, but by providing guidance values for project managers and decision-makers,[10,11] within the context of a regulatory framework (i.e. the WFD).[12]

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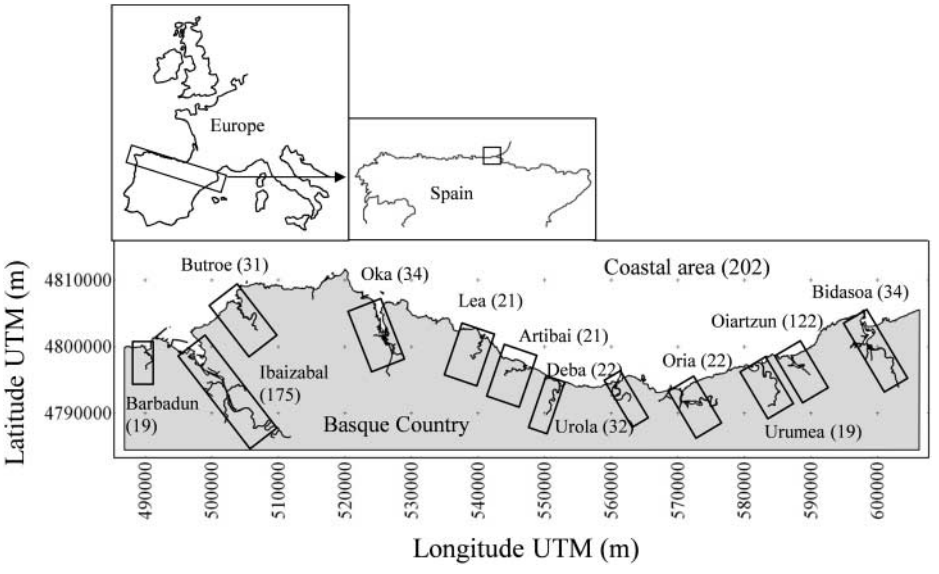

Figure 1. Map of the Basque coast, showing the sampling locations. Note: The number in parentheses indicates the number of samples obtained within each estuary and on the open coast.

International SQG values for metals and organic compounds have been established.[5–8] Nevertheless, according to Chapman,[13] the SQG should be used in the region in which they were developed, to better predict the toxicity of contaminants for each specific coastal environment.[7] Following technical guidance for the derivation of EQS,[2] Menchaca et al.[14] developed empirical regional SQG for metals in coastal and estuarine sediments for the Basque coast (SE Bay of Biscay). Hence, the aim of this article is to calculate the regional SQG for organic compounds (polychlorinated biphenyls [PCBs] and polycyclic aromatic hydrocarbons [PAHs]) for the Basque coast, based upon the chemical, toxicity and benthic biotic index values.

2. Material and methods

A total of 756 sediment samples were utilised in this study, obtained from different estuarine (554) and coastal (202) locations along the Basque coast (Figure 1), between 2002 and 2011; these include both inter- and subtidal (0–65 m water depth) samples. Sediment sampling details are presented in Menchaca et al.[14]

The method followed for the determination of concentrations of PCBs (PCB-28, PCB-52, PCB-101, PCB-118, PCB-138, PCB-153 and PCB-180) and PAHs (acenaphthene, acenaphthylene, antracene, fluorene, naphthalene, phenanthrene, benzo(*a*)antracene, benzo(*a*)pyrene, chrysene, dibenzo(*a*,*h*)antracene, fluoranthene, pyrene, benzo(*e*)pyrene, benzo(*b*)fluorantene, indeno(1,2,3)pyrene, and benzo(*g*,*h*,*i*)perylene) in bulk sediment is explained in Bartolomé et al.[15] Certified IAEA-417 sediment reference material (International Atomic Energy Agency, Austria) was used to check the accuracy of the analytical procedures. Mean recoveries for almost all of the certified PAHs were between 80 and 90%. Indeno(1,2,3)pyrene and benzo(*g*,*h*,*i*)perylene showed lower recovery rates of between 60 and 70%. Mean recoveries for almost all the certified PCBs were between 85 and 110%. Uncertainty related to the results obtained ranged between 25 and 35%. According to Royal Decree 60/2011 (for environmental quality standards in water policy), for PCB and PAH sum calculation, individual congeners below detection limits were

considered as being zero. The range of concentrations for each organic compound is listed in Table 1.

SQG values were calculated from PCB and PAH concentrations, non-normalised and normalised by sediment organic matter (ranging from 0.4 to 36.6%). The organic matter (OM) content was determined as loss of ignition percentage at 450 °C for 5 h. Normalisation on the basis of sediment OM was performed by dividing the dry weight organic compound concentration ($\mu g\,kg^{-1}$) by the percentage of OM in the sediment, expressed as a decimal fraction. Thereafter, in order to allow comparison with SQG values developed in other areas, concentrations were normalised to the 1% total organic carbon (TOC) content, considering that TOC [%] = 0.35*OM [%].[16] Future comparisons with these transformed values have to be undertaken with caution, because the linear relationship was fitted from previous data measured in surficial sediment from the area studied (unpublished data); this may not be appropriate for use in other areas.

The toxicity of the sediments was established using three toxicity tests: (1) the Microtox® or bioluminescence inhibition bioassay; (2) the 48-h embryo–larval toxicity test of the sea urchin *Paracentrotus lividus*; and (3) the 10-day survival test, with amphipods of the genus *Corophium*. Benthic community disturbance was determined by means of the AZTI's Marine Biotic Index (AMBI), using the software available at http://ambi.azti.es. Details on the toxicity tests and AMBI calculation are presented in Menchaca et al.[14] Each of the toxicity and benthic measurements from a sample was ascribed to an 'effect/no effect' descriptor, following the criteria described by Menchaca et al.[14]

The number of determinations for each organic compound (PCBs and PAHs), for toxicity and AMBI, is listed in Table 1. For each sample and organic compound, the data from the four biological effects measured (see above) were considered as different cases. Because of this approach, taking into account that not all the organic compounds and biological effects were measured in all of the sediment samples, the overall number of sediment samples is not coincident with the total data for each organic compound.

SQG values were calculated from the methodology described by Long et al.,[5] the effect range low (ERL) and effect range median (ERM) concentration, and from the methodology described by MacDonald et al.,[6] the threshold effect level (TEL) and probable effect level (PEL). X^2 tests were performed to evaluate whether the increasing effect values with increasing SQG ranges was significant, i.e. if the effect/no effect ratio differed significantly ($\alpha = 0.05$) between each range of the SQG.

3. Results

SQG values, non-normalised and normalised to 1% TOC, are listed in Table 2. Some non-normalised SQG values were not calculated, because organic compound concentrations were below their respective detection limits (ERL and TEL values for total PCBs; ERL values for all PAH congeners except phenanthrene and benzo(*b*)fluorantene), TEL values for low PAH congeners (except for naphthalene and phenanthrene) and ERM and PEL values for acenaphthylene. For the same reason, SQG values normalised to 1% organic carbon values (Table 2) could not be calculated for total PCBs (ERL and TEL values), ERL values for all PAH congeners (except for phenanthrene, indeno(1,2,3)pyrene, benzo(*g*,*h*,*i*)perylene and benzo(*b*)fluoranthene), TEL values for low PAH congeners (except for naphthalene and phenanthrene) and ERM and PEL values for acenaphthylene.

For all of the organic compounds, except for total PCBs, the TEL and PEL values for the Basque coast were slightly higher than the ERL and ERM values, respectively, especially for high molecular weight PAH and for total PAHs. The non-normalised SQG values

Table 1. Some overall statistical values (min, max, mean, SD, 50th, 75th and 95th percentiles) ($\mu g\,kg^{-1}$) and number of toxicity tests (Microtox®, amphipods, sea urchins), AMBI and total data for organic compounds (PCBs and PAHs), generated from 756 estuarine and coastal surface sediment samples within the Basque coast.

Organic compound	Min	Max	Mean	SD	50th	75th	95th	Microtox®	Amphipod	Sea urchin embryo	AMBI	Tota data
Total PCBs[a]	< DL	4,933	92	344	6.45	29	440	93	140	66	724	1,023
Acenaphthene	< 2.00	1,068	17.0	59.1	< 2.00	9,3	95.5	57	98	59	467	681
Acenaphthylene	< 2.00	3,279	22.0	219	< 2.00	5,9	36.8	57	97	59	466	679
Antracene	< 2.00	7,778	160	591	18.0	109	604	93	135	61	516	805
Fluorene	< 1.50	1,682	51.7	134	6.4	44,8	199	82	124	61	496	763
Naphthalene	< 1.80	1,970	87.0	204	14.2	50.7	441	70	111	49	492	722
Phenanthrene	< 1.60	12,363	311	906	50.4	218	1,223	93	140	61	729	1,023
Low molecular weight PAH	< DL	16,928	1,030	2,141	339	1,143	3,886	93	140	61	729	1,023
Benzo(*a*)antracene	< 1.60	14,015	356	992	62.0	294,8	1,456	93	140	61	729	1,023
Benzo(*a*)pyrene	< 1.50	16,861	424	1,401	48.9	305,5	1,594	81	123	45	698	947
Chrysene	< 1.50	13,782	377	1,208	35.3	245,1	1,624	93	140	61	729	1,023
Dibenzo(*a*,*h*)antracene	< 1.80	2,122	106	228	26.6	101,4	416	93	135	61	516	805
Fluoranthene	< 1.50	22,241	556	1,527	84.9	471,8	2,468	93	140	61	729	1,023
Pyrene	< 2.00	45,909	577	2080	95.8	426,7	2,145	93	140	61	729	1,023
High molecular weight PAH	< DL	74,160	2,454	7,092	433	2044,1	10,543	93	140	61	729	1,023
Benzo(*e*)pyrene	< 1.50	9,537	262	647	31.8	223,7	1,339	81	123	45	698	947
Benzo(*b*)fluorantene	< 2.00	17,028	561	1,392	128	507,6	2,023	76	117	49	453	695
Indeno(1,2,3)pyrene	< 2.00	13,993	284	815	32.9	243,6	1,170	81	128	49	729	987
Benzo(*g*,*h*,*i*)perylene	< 2.50	8,652	226	616	22.4	193,6	976	81	128	49	729	987
Total PAH	< DL	136,888	4,352	12,029	671	3459,6	18,356	93	140	61	729	1,023

Note: for sampling locations, see Figure 1.
Note: <DL, below the detection limit. [a]Total PCBs: PCB-28, PCB-52, PCB-101, PCB-118, PCB-138, PCB-153 and PCB-180.

Table 2. Sediment Quality Guidelines (SQG) for organic compounds (PCBs and PAHs) ($\mu g\,kg^{-1}$) calculated for the Basque coast in this study andother studies undertaken elsewhere.

	Basque coast (this study)				Basque Country (this study)*				Norway [6]	OSPAR [16]	USA [3]		USA [4]	
Organic compound	ERL	ERM	TEL	PEL	ERL	ERM	TEL	PEL	PNEC	BAC	ERL	ERM	TEL	PEL
Total PCBs	nd	29	nd	24.6	nd	34.5	nd	30.7	17–190		22.7	180	21.5	189
Acenaphthene	nd	3.09	nd	6.7	nd	1.5	nd	4.5	160–360		16	500	6.71	88.9
Acenaphthylene	nd	nd	nd	nd	nd	nd	nd	nd	33–85		44	640	5.87	128
Antracene	nd	30.1	nd	73	nd	16.0	nd	39.6	31–100	1.8	85.3	1,100	46.9	245
Fluorene	nd	12.9	nd	28	nd	7.9	nd	17.4	260–510		19	540	21.2	144
Naphthalene	nd	28.2	5.2	45	nd	16.0	5.5	31.7	290–1,000		160	2,100	34.6	391
Phenanthrene	3.2	101	14.3	161	1.7	50.9	13.3	97.3	500–1,200	7.3	240	1,500	86.7	544
Low molecular weight PAH	4	164	24.4	285	2.4	88.4	21.2	189			552	3,160	311.7	1,442
Benzo(*a*)antracene	nd	143.3	13	227	nd	64.2	14.2	135	60–90	7.1	261	1,600	74.8	693
Benzo(*a*)pyrene	nd	147	8.9	238	nd	63.8	9.8	125	420–830	8.2	430	1,600	88.8	763
Chrysene	nd	107	9.7	207	nd	52.3	8.9	114	280–280	8.0	384	2,800	108	846
Dibenzo(*a*,*h*)antracene	nd	53.8	4.3	81	nd	23.2	3.9	43.9	590–1,200		63.4	260	6.22	135
Fluoranthene	nd	202	12	343	nd	90.5	14.4	193	170–1,300	14.4	600	5,100	113	1,494
Pyrene	nd	221.5	15.6	333	nd	95.8	18.9	181	280–2,800	11.3	665	2,600	153	1,398
High molecular weight PAH	20.5	922	99.4	1,537	19.8	403	108	813			1,700	9,600	655	6,676
Benzo(*e*)pyrene	nd	116	5.6	163	nd	52.3	5.9	101			–	–	–	–
Benzo(*b*)fluorantene	15.4	294	29.3	408	nd	87.4	5.8	168	240–490		–	–	–	–
Indeno(1,2,3)pyrene	nd	98	3	168	0.5	46.9	6.5	101	47–70	8.3	–	–	–	–
Benzo (*g*,*h*,*i*)perylene	nd	74.9	6.7	137	0.8	41.7	5.1	79.9	21–31	6.9	–	–	–	–
Total PAH	39.5	1,607	172	2,617	36.0	772	197	1,5	2,000–6,000		4,022	44,792	1,684	16,770

Notes: *Non-normalised and normalised to 1% TOC.
BAC, background values by OSPAR; ERL, effect range low; ERM, effect range median; nd, no data; PEL, probable effect level; PNEC, predicted no effect concentrations; TEL, threshold effect level.

are higher than the normalised values, except for total PCBs and for TEL values for naphthalene, benzo(*a*)antracene, benzo(*a*)pyrene, fluoranthene, pyrene, high molecular weight PAH, benzo(*e*)pyrene, indeno(1,2,3)pyrene and total PAHs.

In order to determine the accuracy of the guideline values, the incidence of effects was calculated for the two methodologies.[5] The incidence of the effects associated with the TEL and PEL values, non-normalised by the sediment organic matter, was close to 25% in the minimum effect range (< TEL) for: (1) low and high molecular weight PAH; (2) total PAHs; and (3) some PAH congeners (naphthalene, phenanthrene, pyrene, benzo(*b*)fluorantene). Moveover, they were > 60% in the probable effect range (> PEL) for all organic compounds, non-normalised by the sediment organic matter (Figures 1SM, 2SM, 3SM, 4SM and 5SM, available online only). In contrast, the incidence of the effects associated with TEL and PEL values (normalised by the sediment organic matter) and the incidence of the effects associated with ERL and ERM values (normalised and non-normalised by the sediment organic matter), were not as consistent (Figures 6SM, 7SM and 8SM, available online only).

Moreover, as shown by the X^2 tests, the incidence of the effects increased significantly with increasing SQG ranges, in almost all cases (specially for non-normalised values), except for those outlined below:

- low molecular weight PAH, the ERL value (normalised and non-normalised by the sediment organic matter) and the TEL/PEL values (normalised by the sediment organic matter) (Figure 1SM);
- high molecular weight PAH, the ERL value (non-normalised), the ERM and the PEL value (both normalised) (Figure 2SM);
- total PAHs, the ERM and PEL value (both normalised) (Figure 2SM);
- some PAH congeners:
  (1) non-normalised – naphthalene (the PEL value) (Figure 3SM), phenanthrene (the ERL value) (Figure 3SM), fluoranthene (the ERL value) (Figure 4SM) and benzo(*e*)pyrene (the TEL value) (Figure 5SM);
  (2) normalised – acenaphthene (the PEL value) (Figure 6SM), antracene (both SQG values) (Figure 6SM), naphthalene (both SQG values) (Figure 6SM), phenanthrene (the TEL and ERL values) (Figure 6SM), benzo(*a*)antracene (the ERM value) (Figure 7SM), benzo(*a*)pyrene (the TEL/PEL value) (Figure 7SM), chrysene (the TEL value) (Figure 8SM), dibenzo(*a*,*h*)antracene (the PEL value) (Figure 7SM), fluoranthene (the TEL/PEL value) (Figure 7SM), pyrene (the TEL value) (Figure 7SM), benzo(*e*)pyrene and benzo(*b*)fluorantene (the TEL value) (Figure 8SM), indeno(1,2,3)pyrene (the ERM and PEL value) (Figure 8SM) and benzo(*g*,*h*,*i*)perylene (the ERL and TEL/PEL value) (Figure 8SM).

4. Discussion

Despite the uncertainties involved in the SQG approach,[17] some authors[18] have recommended the use of empirical SQG values to assess overall environmental quality and, if necessary, to set remediation goals, because of the following advantages described previously in Menchaca *et al.* [14]: (1) they are calculated from a large database of correlative effects, using field-collected sediments, i.e. that available for the Basque coast; (2) they can be applied within a regulatory framework, i.e. the WFD or others, serving as scientific advisory values; and (3) they show a wide geographical application (in our case, probably for the same ecoregion, within the Bay of Biscay). Hence, both empirical guideline approaches (especially for non-normalised values) calculated in

this study for the sediments of the Basque coast, showed good accuracy due to the significant increasing effect values with increasing SQG ranges, in almost all of the cases investigated.

Background levels for naturally occurring substances (i.e. PAHs) and levels close to zero for man-made synthetic substances (i.e. PCBs) are used in assessing the WFD physicochemical status (representing the boundary between 'high' and 'good').[1] Hence, in the case of PAHs, it may be expected that background values derived by OSPAR[19] should be lower than the SQG, i.e. with no effect on the biota. On the one hand, in our investigation, the TEL values for selected PAHs (non-normalised) were higher than background values, except for indeno(1,2,3)pyrene (Table 2). However, the ERL values could not be compared with background values, because the concentrations were below their respective detection limits; these were lower than their respective background values, except for phenanthrene (Table 2). Clearly, most of these apparent incoherencies arise from differences in the approaches and methodologies used in the background and SQG calculations. In the same way, the ERL values proposed by Webster et al.,[20] *i.e.* benzo(*a*)antracene, benzo(*g*,*h*,*i*)perylene and indeno(1,2,3)pyrene, are lower than the background values, recognising that those ERL are not recommended for use in sediment assessments. At this point, the ERL values could not be considered at the threshold of sediment toxicity.[21] On the other hand, the ERM and PEL values for all organic compounds were higher than the background values (Table 2), with ratios ranging from 11 to 20 and from 20 to 41, respectively. Furthermore, taking into account that the incidence of effect in the ranges above the ERM and PEL was close to 70%, the ERM and PEL values could be considered as a threshold above which effects occur frequently.

In this study, normalisation by sediment organic matter did not result in any improvement in accuracy. In order to compare the Basque coast SQG values obtained in this work, with those calculated for other areas, international SQG values are listed in Table 2. The non-normalised SQG values calculated for this study are within the range of SQG values determined for different areas, i.e. those developed for Norway[8] (total PCBs, antracene, fluoranthene, pyrene, benzo(*b*)fluoranthene, total PAHs) (Table 2). Furthermore, comparing the values calculated here with the available ecotoxicological data (EC_{10}/EC_{50}/LOEC), the Basque coast SQG values are within the range calculated for sea urchins by Bellas et al.[22] for naphthalene, phenanthrene, pyrene and fluoranthene.

As commented upon in the Introduction, 'good' physicochemical status can be considered when pollutant concentrations lie between the background level and the EQS, within the WFD. Directive 2013/39/EU establishes the EQS for priority substances (such as anthracene, fluoranthene, naphthalene, benzo(*a*)pyrene, benzo(*b*)fluoranthene, benzo(*g*,*h*,*i*) perylene, indeno(1,2,3)pyrene) in waters and biota, but not for sediments. Nowadays, international SQG for organic compounds, such as those proposed by Long et al.[5] and MacDonald et al.,[6] are used frequently as the EQS in investigative monitoring for the ecological status assessment within the WFD.[1,23] Hence, the regional SQG values developed in the present study provide guidance values for project managers and decision-makers,[10] in assessing chemical and physicochemical status, to be used within the WFD.

Supplementary material

Supplementary material relating to this article is available online.

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