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Report

Studies on phosphorus removal from fresh water and sea water By commercial sorbents

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1. Introduction

Dissolved orthophosphate is the predominating phosphorus species in fresh and sea water. Typical concentrations in natural uncontaminated fresh and sea water range from 5 to 30 $\mu\text{g/L}$ referring to phosphorus (Grobkopf 1998).

In aquaria these concentrations can be dramatically exceeded due to increased supply with phosphate combined with insufficient fresh water exchange.

Main sources for phosphorus are the fish food, esp. when containing proteins (mussel, krill), the excreted food and dying organisms, which contain phosphate in their cell membranes. As a consequence an increased growth of algae can be observed. Furthermore, increased phosphate concentrations influence the synthesis of limestone due to precipitation of calcium phosphates and therefore complicate the growth of corals.

In most cases the potential of water plants for removing dissolved phosphorus is not sufficient to guarantee the required water quality. Frequent water replacement may lead to satisfying phosphorus concentration but is a very expensive solution.

The irreversible removal of phosphorus from the water cycle is possible by using solid adsorption media. The ideal media has a high capacity and a high specificity regarding phosphate and should not influence other water parameters. '

2. Tested sorbents

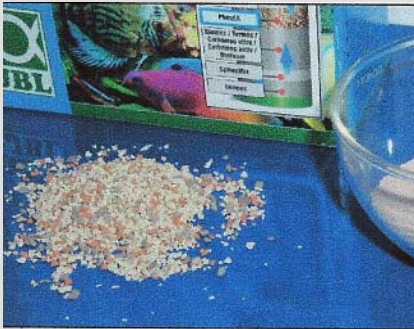

Several commercial media are available for removal of phosphate from fresh and sea water which are recommended to be used in the circulation filters of the tanks. The studied media are iron-based (RowaPhos) or aluminum-based (ElimiPhos), some in the form of zeolite (AntiPhos, PhosEx) or ceramics (Phosphate Sponge), **Table 1** gives a summary of the sorbent's characteristics.

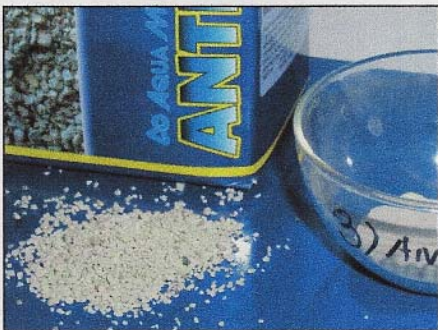
The following studies with 5 sorbents were carried out according to our offer dated June 6th 2002 :

1. phosphate sorption isotherms for concentrations smaller than 1 mg/L P for Berlin tap water and artificial sea water,
2. elution studies with subsequent determination of dissolved organic carbon (DOC), pH, conductivity, heavy metals (Cu, Pb, Zn, Ni, Cd) and elements Al, Fe, Mn.

Table 1: Characteristics of the tested sorbents

Sorbent	RowaPhos	Elimi Phos
Producer	Weil Industrieanlagen GmbH Heinrich-Hasemeier-Str. 33 D-49076 Osnabrück	Tropic Marin Tropic Marin 44 Center Street Montague, MA 01351 (USA)
		Dr. Biener GmbH Steinäcker Str. 3-5 D-36367 Wartenberg
		
Specifications (acc. to producer)	pure β -FeOOH	granulated material
add. information of producer	use 100 ml RowaPhos for 100-200 L sea water	100 g for 200 L water, add. removal of organic substances

Sorbent	Phos Ex	Phosphate Sponge
Producer	JBL GmbH & Co. KG Postfach 17 D-69137 Neuhausen (USA)	Kent Marine, Inc, 1100 Northpoint Parkway Acworth, Georgia 30102
		
Specifications; (acc. to producer)	natural zeolite, specially treated	ceramics
add. Information use, of producer	11kg PhosEx binds 209 PO_4^{3-} 5-6 months efficient, PO_4^{3-} is reduced from 10 mg/L to 1 mg/L, long contact time recommended	rinse with d.i. water before sufficient for 120 gal (454L)

Sorbent	Anti Phos
Producer	Aqua Medic Gewerbepark 24 D-49143 Bissendorf
	
Specifications (acc. to producer)	natural zeolite granulate material, 200 m ² /g surface, chemically modified, porous base
add. information of producer	1000 ml AntiPhos for 200 L water

3. Methods

3.1 Sorbent preparation

For determination of sorption isotherms, all sorbents were ground in a mortar by hand and sieved to a fraction $< 63 \mu\text{m}$,

Because dried RowaPhos will lose its capacity, a special preparation process was developed for this sorbent:

- Rinsing with Millipore -water,
- Adjustment of pH (8) with NaOH,
- Drying at room-temperature for 16 h (water content -22 %),
- Grinding per mortar and sieving to the fraction $< 63 \mu\text{m}$.

3.2 Sea water preparation

For first studies the commercial salt "Tropic Marin" was used for sea water preparation (33 g/L Millipore -water). After one day storing this water showed a high turbidity due to precipitation processes. Therefore "Tropic Marin" was not usable for analytical purposes and was replaced by a salt produced by Dr. Biener GmbH (36367 Wartenberg, Germany). This water showed a better behavior during subsequent analytics and no precipitation was observed during the whole period. The density was determined with 1.017 kg/m^3 at 28°C .

3.3 Isotherms

Six isotherm points were determined for each sorbent at the original pH of sea water (pH 8.0) and tap water (pH 8.1).

To evaluate adsorption isotherms, the 'method of adding different quantities of sorbent (m) to solution volumes (L ; L/m variable) of the same initial concentration ($c_0 = 1 \text{ mg/L P}$) was used. The initial concentration of 1 mg/L P was produced by dilution of a phosphorus stock solution ($c = 1 \text{ g/L P}$) which

buffered at the original pH of the sea water (3.7 parts KH_2PO_4 and 96.3 parts $\text{Na}_2\text{HPO}_4 \cdot 2\text{H}_2\text{O}$ in Millipore - water).

Defined quantities of sorbent ($m = 5 - 800$ mg dry matter) were added to several glass bottles containing defined volumes of sea water/tap water ($L = 200$ ml). The flasks were shaken for 96 h at 20°C on a linear shaker and subsequently filtered through 0.45 micron cellulose nitrate filter (Sartorius). The pH was measured immediately. The filtrates were stored for P-analytics at 10°C .

The equilibrium loading q in dependence on the reached equilibrium concentration c was calculated using the mass balance of the system:

$$q = L/M (c_0 - c_{eq}) \quad (1)$$

with q : solid-phase concentration [mg/g DM], c_0 : initial liquid-phase concentration [mg/L], c_{eq} : liquid-phase concentration [mg/L] at equilibrium, L : liquid volume [L] and m : mass of sorbent [g DM].

The calculated loadings are related on the dry mass of each sorbent (**Table 2:**).

Table 2:

Water content and dry mass of all sorbents. For RowaPhos for the pretreated material according to chapter 3.1 and the original material.

Sorbent	RowaPhos Sponge	AntiPhos	ElimiPhos	Phos Ex	Phosphate
Water Content (%)	21.0 (pre-treated) 54.0 (original)	5.2	11.1	2.3	11.6
Dry Mass (%)	79.0 (pre-treated) 46.0 (original)	94.8	88.9	97.7	88.4

3.4 Elution studies

The elution studies were performed according to DIN-standard 38414 [1]. 50g of the original sorbent (moist mass) were shaken with 0.5 L water for 24 h at 20°C.

For the parameters pH, conductivity, dissolved organic carbon (DOC) and UV absorbance (SAC_{254}), the elution was performed with Millipore water in order to avoid buffering and interaction with water components. It has to be emphasized that this elution procedure might not be comparable to the conditions in an aquarium in which the water is well buffered, but helps to understand the composition of the media.

Since most heavy metals are known as mobile cations at low pH, the pH is believed to be the decisive parameter during elution. pH values lower than 6 or higher than 9 might lead to an increased dissolving of heavy metals. Therefore, the elution was tested with tap water. Since the pH was kept constant between 6.5 and 8.5 (by adding HCl/NaOH), this procedure allows reasonable comparison between the different media. It can be assumed that in most cases the pH in an aquarium should be within this range.

Subsequent to elution, the samples were filtered and stored for analytics,

4. Analytics

4.1 Sample preparation

Samples for determination of DOC, SAC₂₅₄ and Orthophosphate were filtered through 0.45 micron cellulose-nitrate-filter (Sartorius). Samples were kept 10°C until analyses.

For exemption from P all glassware were stored in an acid bath (HCl 10%) overnight and rinsed with Millipore water.

4.2 Orthophosphate

For determination of orthophosphate a flow injection analyzer (FIAstar 5000, Foss Tecator) was used according to ISO/DIS 15681:2001 part 1. The analysis bases on the formation of heteropolyacids in the presence of phosphate and molybdate ions in acidic solution, which are reduced in a second step by tin(II)chloride to a blue colored molybdate complex. The detector measures two wavelengths (720 nm, 1000 nm) simultaneously. The reference wavelengths of 1000 nm is used to compensate optical and electronic fluctuations. The signal is evaluated using the peak height. The activated drift control excludes peaks with shape abnormality (tailing, fronting). Each value is an average value of three measurements. All samples were measured at room temperature.

Calibration was performed with sea water and tap water standards, respectively. Measuring ranges of 0 – 100 micro g/L and 0 – 500 micro g/L P were utilized. The lowest standard was 5 micro g/L for both calibration ranges. Daily fluctuations were compensated by using daily factors determined with the highest standard of each calibration (100 and 500 *micro g/L* P). As carrier were used Millipore-water for isothermms in tap water and salt solution (33 g/L NaG!) for isotherms in sea water.

The detection limit is specified with 0.5 micro g/L P with a relative standard deviation of 0.7% related on a 100 micro g/L P standard (Foss Tecator 2001).

4.3 Dissolved organic carbon

Dissolved organic carbon (DOC) was analyzed undiluted using a LiquiTOC analyser with autosampler by wet oxidation. After acidification with phosphoric acid and removal of inorganic carbon the sample is oxidized with peroxodisulfate, oxygen and UV light. As oxidation product CO₂ is measured using NDIR detection. Calibration was performed with phthalate solutions ranging from 0.25 to 10 mg/L C. The detection limit is specified with 0.40 mg/L with a standard deviation of 0.27 mg/L.

4.4 Spectral absorption coefficient at 254 nm (SAC₂₅₄)

Spectral absorption coefficients SAC at 254 nm [1/m] were determined using a Lambda 12 UV/VIS-spectrophotometer (Perkin-Elmer) and cuvettes of 1 cm length. Because of the linear correlation between the SAC₂₅₄ and the DOC in natural waters, the SAC₂₅₄ is used as a control measurement for the DOC values.

4.5 pH, conductivity, turbidity

The pH was measured by a WTW pH 340-meter using a Sentix 41-electrode (WTW). Calibration was performed with technical standards (WTW). Conductivity was measured using a WTW LF 323-A conductivity meter. Turbidity was determined using a Dr. Lange LTP 5 turbidity meter measuring stray light at 90°.

4.6 ICP-MS, AAS

Metal ions were measured using a Quadrupol-ICP-MS ELAN 6000 (Perkin Elmer). As detector a mass spectrometer with CEM was used. 0.1 ml Sc-solution per 10 ml sample were added in order to compensate matrix effects. Calibration was performed using a multi element standard IV (Merck). For further elution studies, metal ions were measured using flame atomic absorption spectroscopy (SpectrAA 400, Varian).

5. Results

5.1 Results of isotherm studies

The adsorption of phosphate on metal oxide surfaces has been well studied by numerous authors. Phosphate is regarded as a specific adsorbing ion, which means that phosphate can adsorb at pH values higher than the pH_{pzc} (point of zero charge) by the formation of complexes. Sigg and Stumm (1981) describe the formation of inner spheric complexes on metal oxide surfaces. Persson et al. (1996) suggest the formation of a mono dentate complex for sorption of phosphate on goethite. Rietra et al. (2001) additionally point on the role of calcium for these sorption processes

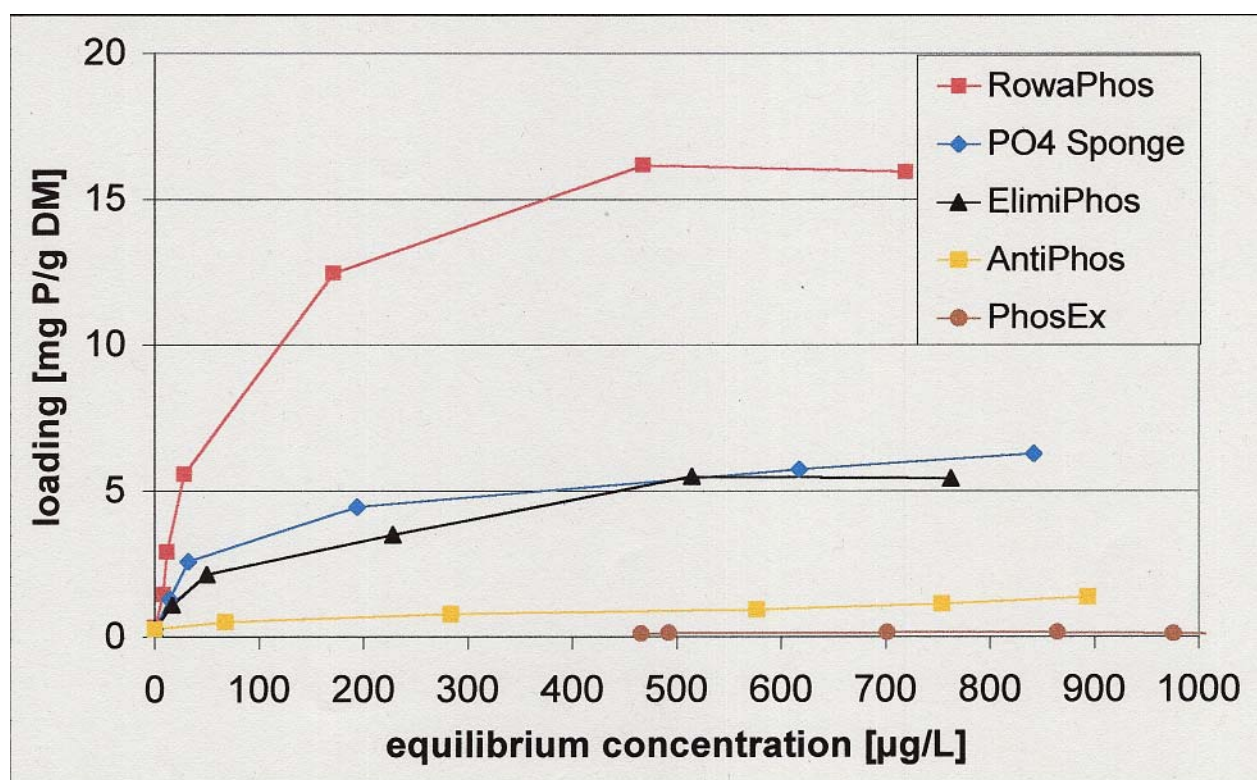


Figure 1

Phosphate-isotherms, Berlin tap water, pH 8
(20°C, 96h shaking time, $c_0=1 \text{ mg/L P}$)

In Figure 1 phosphate isotherms in Berlin tap water are shown. The isotherms in sea water (Figure 2) show a similar trend as in tap water. The equilibrium loadings are somewhat smaller than those in tap water but still in the same magnitude.

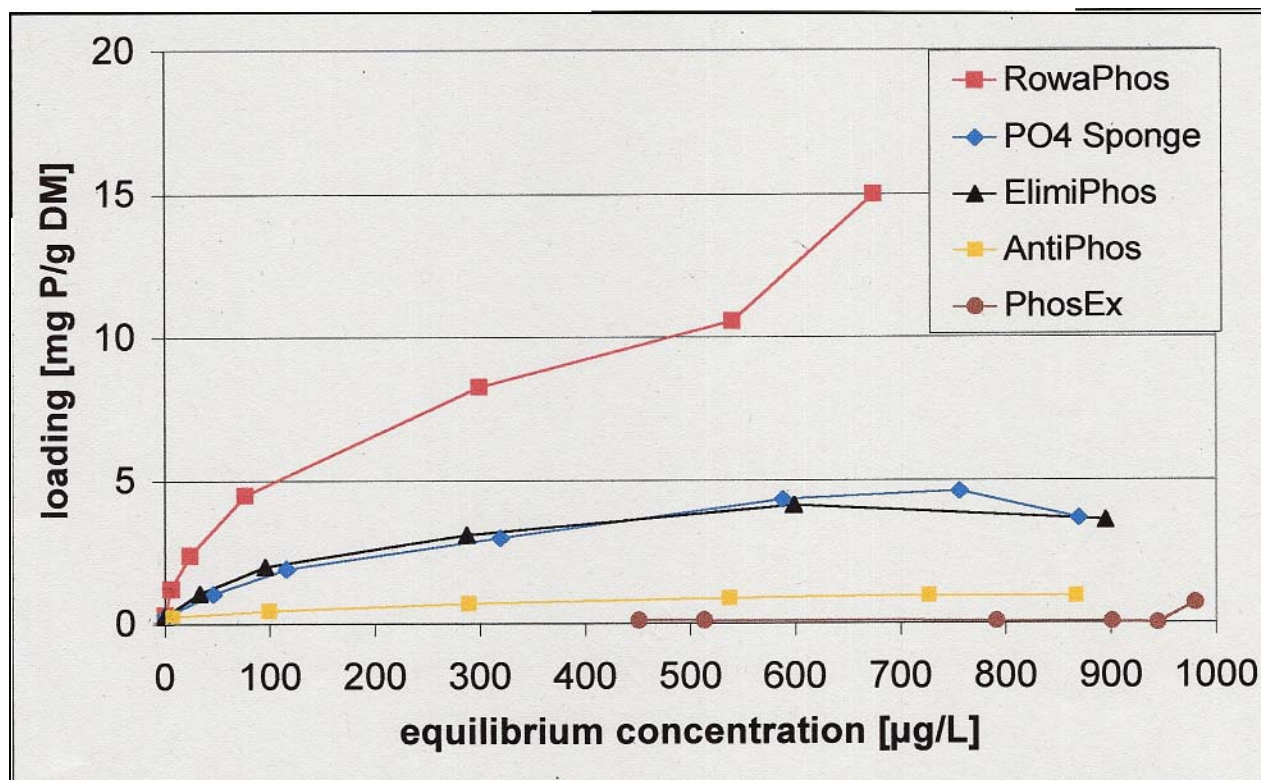


Figure 2

Phosphate-isotherms, sea water (Dr. Biener), pH 8,
(20°C, 96h shaking time, $c_0=1 \text{ mg/L P}$)

With RowaPhos the highest loadings were achieved. The reached maximum loading of around 15 *mg/g* agrees with earlier studies at the Department of Water Quality Control in pure systems (Driehaus 1994).

The isotherms of Phosphate Sponge and ElimiPhos run remarkable parallel for both waters. Therefore, it is assumed that these media consist of identical structures, supposedly on the basis of aluminum oxide. Altogether they showed a moderate adsorption performance with loadings around 5 *mg/g*

Though it was possible to remove all phosphate from the solution with AntiPhos (which means that equilibrium concentration of 0 mg/L is reachable), the achieved loadings are comparably low (smaller than 1 mg/g). The isotherm's shapes show only weak affinity towards phosphate.

The most eye-catching characteristics showed the sorbent PhosEx. Even with the highest sorbent dosage the equilibrium concentration does not drop below 450 micro g/L. P. The achieved loadings are very low or almost zero.

5.2 Description with the Langmuir isotherm equation

For mathematical description of the isotherms and a better comparison, adaptation was performed using the Langmuir isotherm equation:

$$q = q_m \cdot \frac{K_L \cdot c}{1 + K_L \cdot c} \quad (2)$$

With q : loading [mg/g DM], q_m : [mg/g DM], c : liquid-phase concentration (mg/L) at equilibrium, K_L : Langmuir constant [L/mg]

This equation was developed for the description of the adsorption of gases on solid surfaces (Langmuir, 1918). q_m characterizes the maximum capacity (monomolecular loading) of the sorbent, K_L the affinity which can be seen as the slope of the isotherm curve. Equation (2) can be transformed into the linear function (3)

$$c / q = (1 / q_m \cdot K_L) + (1 / q_m) \cdot c \quad (3)$$

The two constants of the Langmuir isotherm equation (K_L , q_m) can be determined from isotherm data by linear regression. In general, this method gives good results for the isotherm points at higher equilibrium concentrations and leads to an accurate description of q_m .

The adaptation was performed for the media RowaPhos, ElimiPhos and Phosphate Sponge. The regression coefficients varied from 0.94 to 0.99. Due to the poor adsorption characteristics of AntiPhos and PhosEx, a reasonable adaptation was not possible for these two media.

The obtained isotherms are shown in Figure 3 (tap water) and Figure 4 (sea water). For adaptation for RowaPhos in sea water, the isotherm points for the, highest L/m ratio were excluded. These points might have been influenced by precipitation, most likely of calcium phosphate.

For practically removing phosphorus from aquaria, not the maximum capacity q_m might be of relevance, but the reachable loading in the range of smaller phosphorus concentrations. For this reason, the achieved loadings at equilibrium concentrations of 50 and 100 *micro g/L* P were evaluated and compared. The higher are these loadings, the steeper is the Isotherm curve and the more efficient is the P removal in the small concentration range. The obtained parameters of adaptation are shown in **Table 3**.

Table 3:

Maximum capacity q_m and capacity at equilibrium concentrations of 50 and 100 micro g/L P (mg P/g DM), KL (L/mg) and R^2 for Berlin tap water and sea water according to the Langmuir isotherm equation.

		RowaPhos	ElimiPhos	Phosphate Sponge
Berlin tap water	q_m (mg P/g DM)	17.3	5.9	5.8
	q_{50} (mg P/g DM)	8.5	2.5	3.6
	q_{100} (mg P/g DM)	11.4	3.5	4.4
	correlation coefficient R^2	0.99	0.97	0.98
sea water	q_m (mg P/g DM)	11.3	3.6	4.6
	q_{50} (mg P/g DM)	4.8	2.0	1.5
	q_{100} (mg P/g DM)	6.8	2.6	2.3
	correlation coefficient R^2	0.96	0.96	0.94

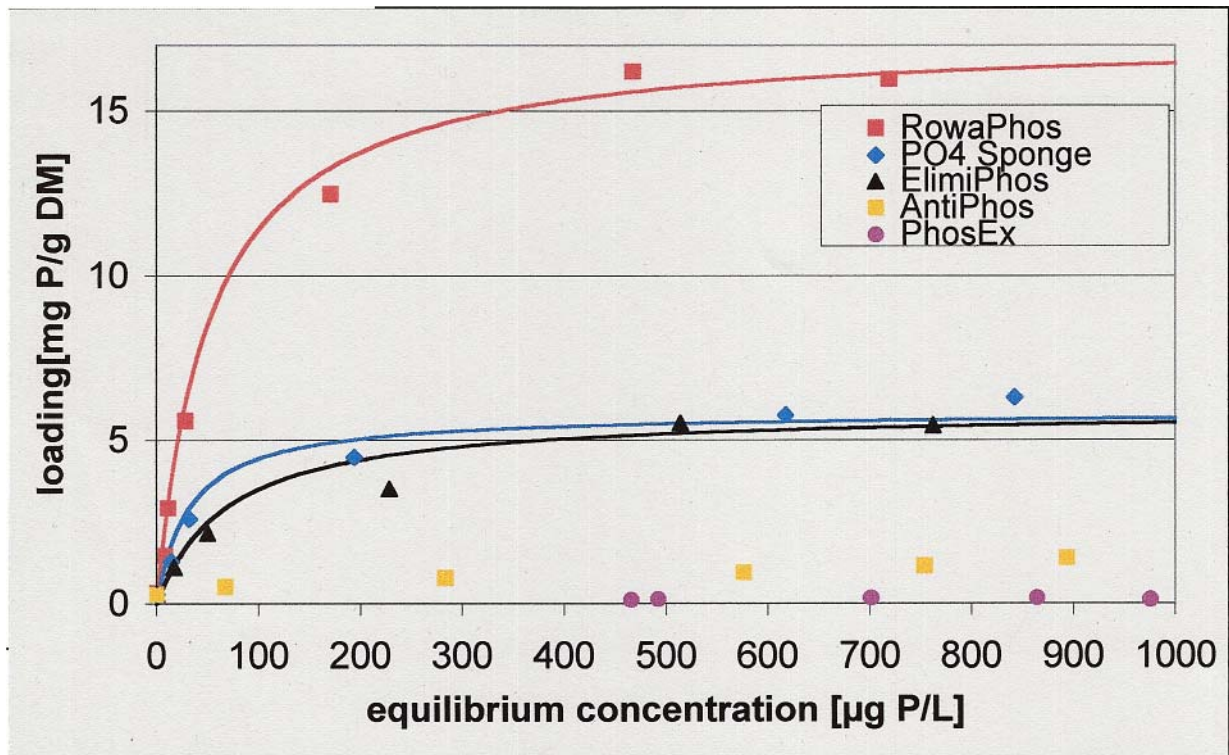


Figure 3 Phosphate isotherms (acc. to Langmuir equation), Berlin tap water, pH 8, (20°C, 96h shaking time, $c_o = 1 \text{ mg/L P}$)

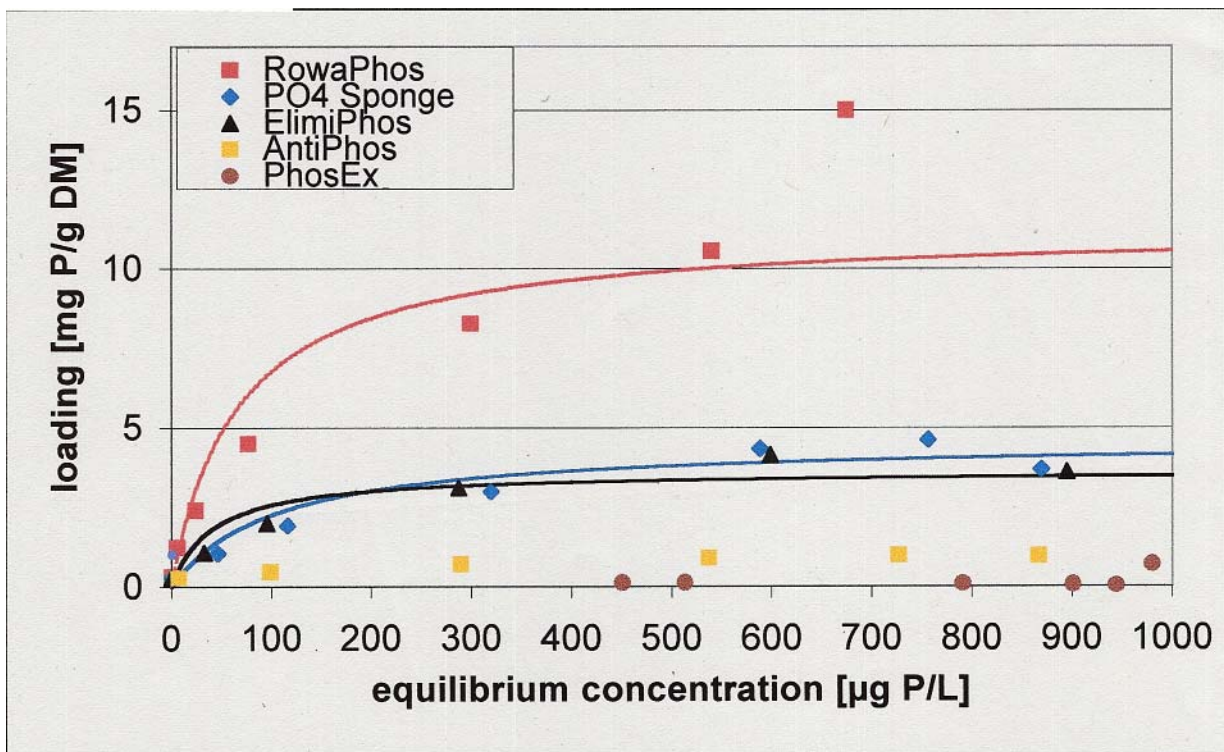


Figure 4 Phosphate isotherms (acc. to Langmuir equation), sea water (Dr. Biener), pH 8 (20°C, 96h shaking time, $c_o = 1 \text{ mg/L P}$)

For the 3 media a decrease of capacity in sea water could be observed compared to tap water ranging from 17 to 35% of the maximum capacity. Therefore, even with a high excess of competing ions (chloride, sulfate) in sea water, only a relatively small decline in loading was found. Consequently, phosphate seems to adsorb mainly by specific complex formation for all tested media. The sorbent surface should be negatively charged at pH 8. As a consequence, the non-specific adsorbing ions like chloride and sulfate do not show strong competition on the sorption of phosphate. Likewise, effects by co-sorption of Calcium could not be observed since Calcium was excessively present for both tap water and sea water.

RowaPhos showed in both waters the highest maximum capacity (q_m). The q_m for ElimiPhos and Phosphate Sponge were around one third of RowaPhos for both tap water and sea water.

With regard to q_{50} and q_{100} , again the performance of RowaPhos was the best with around 5 mg P/g DM. For ElimiPhos and Phosphate Sponge, q_{50} and q_{100} ranged between 30 and 40% of the values for RowaPhos.

For the practical application in a adsorption filter it might be relevant how much phosphorus is removable with regard to a given adsorption filter volume. In **Table 4** the mass of phosphorus that can be removed from tap water in a adsorption filter of 1 L volume is calculated for all sorbents using the respective maximum loadings (chapter 5.1 and 5.2) and the bulk densities.

Table 4:

Removable phosphorus in a 1L adsorption filter

	Rowa Phos	Elimi-Phos	Phos. Sponge	AntiPhos	PhosEx
moist bulk density (g/L)	1200	1270	597	970	970
dry bulk density (g/L)	560	1130	528	919	948
maximum loading (mg/g DM) in tap water	17.3	5.9	5.8	1.4	0.17
mass of removable phosphorus in tap water (g)	9.7	6.67	3.06	1.29	0.16
maximum loading (mg/g DM) in sea water	11.3	3.6	4.6	0.96	0.11
mass of removable phosphorus in sea water (g)	6.3	4.1	2.4	0.88	0.10

5.3 Results of elution studies

In **Table 5** the results of elution with Millipore water are shown. With regard to conductivity, a remarkable shift was observed for AntiPhos up to 1500 micro siemens/cm indicating increased dilution of inorganic salts. All other samples ended up with a conductivity between 250 and 350 micro siemens/cm.

Table 5: Results of elution studies (elution with millipore water, 100 g moist mass per L, 24 h shaking time, 20°C)					
	Rowa Phos	Elimi-Phos	Phosphate Sponge	Anti Phos	PhosEx
pH	4.8	8.7	10.0	4.9	5.4
Conductivity ($\mu\text{S/cm}$)	259	341	273	1508	336
DOC (mg/L)	n.d.	2.64	2.12	1.23	1.75
SAC₂₅₄ (1/m)	0.23	1.96	2.32	0.68	1.19

RowaPhos and AntiPhos cause a pH shift from pH 5.4 (Millipore) down to 4.8 and 4.9, respectively. ElimiPhos and Phosphate Sponge are alkaline media, causing a pH shift from pH 5.4 (Millipore) up to 8.7 and 10, respectively. PhosEx is the only medium that did not cause any shift in pH.

Generally, the DOC and SAC₂₅₄ values show a parallel behavior. For ElimiPhos and Phosphate Sponge an increased DOC (around 2.5mg/L) was measured. As found during the isotherm studies and the pH values (s.a.), these two media again show a parallel behaviour, which confirms the assumption, that these media have similar structures and are supposedly produced in a comparable procedure. AntiPhos and PhosEx showed a moderate elution with 1.2 and 1.7 mg/L DOC. For RowaPhos only little elution of organics was observed. In general the found DOC values are comparatively low when considering the high mass to volume ratio used.

Table 6 gives the results of elution with tap water at pH values between 6.5 and 9.5.

Table 6: Results of elution studies (elution with tap water ,pH 7-8, 100g moist mass per L, 24 h shaking time, 20°C)

	Rowa Phos	Elimi-Phos	Phosphate Sponge	Anti-Phos	PhosEx
pH	7.0	7.4	7.5	7.8	7.4
Cu (mg/L)	<0.01	<0.01	<0.01	<0.01	<0.01
Pb (mg/L)	<0.01	<0.01	<0.01	<0.01	<0.01
Zn (mg/L)	<0.01	<0.01	<0.01	<0.01	<0.01
Ni (mg/L)	<0.01	<0.01	0.01	<0.01	<0.01
Cd (mg/L)	<0.01	<0.01	<0.01	<0.01	<0.01
Al (mg/L)	<0.01	<0.01	0.01	0.4	0.3
Fe (mg/L)	<0.01	<0.01	<0.01	<0.01	<0.01
Mn (mg/L)	0.01	<0.01	<0.01	0.53	0.29

For all sorbents, no elution of the harmful heavy metals Cu, Pb, Zn, Ni and Cd was observed.

In general, dissolution of aluminum and iron is strongly connected with the respective pH. Aluminum is almost insoluble at pH 6.0 to 7.5, iron at pH 6 to 10, but the solubility increases again at pH values below or higher. For all media, no significant iron dissolution was observed. For Phosphate Sponge, AntiPhos and PhosEx, only slightly increased values for aluminum were found. This might indicate that these media contain significant amounts of aluminum.

Manganese was found in relative small concentrations for AntiPhos (0.53 mg/L) and PhosEx (0.29 mg/L}. According to the WHO guidelines, manganese is regarded as one of the least toxic elements. The WHO drinking water standard is 0.5 mg/L. Considering the high mass/volume ratio used, the found manganese concentrations are not regarded as dangerous.

6. Conclusions

For assessment of the studied media the capacity at higher and low equilibrium concentrations and the price should be considered. These parameters strongly depend on the initial P concentration and the target concentration. Additionally, potential harmful impacts on the aquaristic flora and fauna should be taken into account.

For the range of low P concentration, the iron-based medium RowaPhos shows the best sorption characteristics in tap and sea water. This medium is recommendable for low P target concentrations since it showed the highest q_{50} and q_{100} values.

ElimiPhos and Phosphate Sponge showed very similar sorption characteristics, but the price for phosphate sponge is significantly lower. It is assumed that these media mainly consist of aluminum oxide.

AntiPhos lies in the same price range but showed significant lower loadings. This might be the reason why the producer recommends for application a 10 times higher amount for 200 L sea water than for all other media.

PhosEx has the lowest price of all media and also the lowest performance in terms of loading and affinity. Removal of phosphate down to concentration lower than 500 *micro g/L* was not possible. High sorbent dosages might be necessary to achieve a low P concentration in aquaria, which might increase costs for the process design (larger adsorbers etc. The producer specifies that PhosEx would reduce the phosphate concentration from 10 mg/L to 1 mg/L (concentration probably given as phosphate). Summarizing, the zeolite based media AntiPhos and PhosEx are not recommendable for the range of low P-concentrations. It is not likely that these media show a better performance at higher P-concentrations.

For all media an impact on the pH of Millipore water was found. Although sea water will be buffering, it is recommendable to rinse esp. RowaPhos, AntiPhos and Phosphate

Sponge before application. It is believed that all tested media contain heavy metals; therefore the pH should be kept between 7.0 and 8.5 to prevent elution. AntiPhos showed additionally increased conductivity after elution.

For the application in a fixed bed adsorber not only the sorption equilibrium is of interest but also the sorption kinetics. This has to be considered for the design layout, esp. regarding the contact time in the filter. More advanced and expensive testing in pilot or full scale would be necessary to assess these parameters.

Considering the German drinking water standards (**Table 7**), the found values for Cu, Pb, Zn and Ni are below these standards. For cadmium, the standard is lower than the detection limit. The PhosEx and AntiPhos eluates showed a slightly increased Mn value, which might be connected with the indefinite composition of these media. For assessment of the eluted metal concentrations, the applied mass of sorbent in relation to the water volume used in the sea water tank has to be considered.

According to the producer's specifications (Table 1), the recommended mass/volume ratios are around 100 times smaller than those used in the elution studies. Therefore, the measured concentrations can be roughly divided by 100 in order to obtain the effective - concentrations during application. Consequently, all metal concentrations measured after elution would be below these standards. However, some special fishes may be more sensitive to heavy metals than humans.

Table 7:

German drinking water standards according to the Trinkwasser- Verordnung (2001)

Metal	Cu	Pb	Zn	Ni	Cd	Al	Fe	Mn
Concentration (mg/L)	2	0.01	-	0.02	0.005	0.2	0.2	0.05

7. Literature

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