

Changes in water biostability levels during an ion-exchange process using Miex-DOC resin

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ABSTRACT

This article presents the results of studies on organic and non-organic substrate removal by the ion-exchange process using a Miex-DOC resin in surface and infiltration waters for human consumption. Studies have shown that receptivity to ion-exchange decreases as follows: nitrates > phosphate > biodegradable dissolved organic carbon. After the ion-exchange process, the limiting factors in regrowth of micro-organisms in the majority of water samples were found to be the concentrations of all analyzed substrates. A dose of 20 mL/L of resin with a contact time of 30 minutes was found to be sufficient for substrate elimination, regardless of water type. Increasing the resin dosage did not significantly improve substrate removal effectiveness. The ion-exchange process using the Miex-DOC resin allows for an effective elimination of organic and inorganic substrates from water. However, achieving biostability of such water is only possible in cases of limited organic carbon contamination of water.

Key words | biodegradable dissolved organic carbon, Miex-DOC ion-exchange resin, substrates, water biostability, water treatment

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INTRODUCTION

Insufficiently treated water introduced into the water distribution network has been shown to be the main cause of secondary micro-organism development in the distribution network, resulting in a lack of biostability (Srinivasan & Harrington 2007). A consequence of micro-organism regrowth is water contamination during its transport to the end user, not only with micro-organisms, including pathogens, but also with metabolic products from these micro-organisms. Therefore, due to the hazard to human health as well as consequences such as fouling of pipe, biocorrosion, taste and color, it is necessary to ensure biostability in treated water. In effect, it is necessary to not only remove micro-organisms from water, but to also decrease the content of substrates crucial to their development. It is necessary to limit the content of organic substrates, i.e. biodegradable dissolved organic carbon (BDOC) and inorganic substrates, i.e. phosphorus (PO_4^{3-}) and nitrogen ($\text{NO}_3^- + \text{NO}_2^- + \text{NH}_4^+$) (Bucheli-Witschel *et al.* 2012).

Conventional water treatment systems (coagulation, sedimentation, filtration and disinfection) ensure an effective elimination of precursors of disinfection by-products (Ødegaard *et al.* 2010) and phosphates (Jianga *et al.* 2011). Unfortunately BDOC removal effectiveness is often insufficient to limit regrowth of micro-organism development in the water delivery system (Stackelberg *et al.* 2007). Owing to this, conventional water treatment systems include indirect oxidation (most often ozonation), and biofiltration, which serves to biodegrade BDOC (Simon *et al.* 2013) and to assimilate non-organic substrates. Studies conducted in real-life conditions (Świdarska-Bróz & Wolska 2012) have shown that even biofiltration preceded by ozonation does not always ensure a sufficient elimination of biodegradable organic substances within limits of biostability for BDOC concentration equal to 0.25 gC/m^3 .

Therefore, relative to ensuring treated water biostability, it seems justified to use the ion-exchange process on magnetic macroporous anion-exchange resin Miex-DOC.

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Current studies have shown that the Miex-DOC resin ensures a significant reduction in BDOC and total halo-organic formation potential (Graf *et al.* 2014). It has also been shown (Kaewsuk & Seo 2011; Zhang *et al.* 2014) that low molecular weight substances are most effectively removed by the ion-exchange process with this resin, which would indicate the effectiveness of the Miex-DOC process in removing biodegradable substances, which are dominated by simple compounds of a low molecular weight (Nkambule *et al.* 2011).

Additional studies by Zhang *et al.* (2012) have shown a high effectiveness of ion exchange using the Miex-DOC resin in removing non-organic anions from water. This suggests the possibility of removing both organic and non-organic substrates during water pre-treatment. Consequently, using Miex-DOC resin could lead to a reduction in water treatment system complexity.

Previous research focused on the possibility of DOC removal and UV absorbance decrease and therefore removal of disinfection by-product precursors. Unfortunately few studies concentrated on organic substrate removal. In addition, studies concerning inorganic substances removal efficiency concentrated on the possibility of decreasing water corrosivity or hardness but not on limiting non-organic substrates.

Limited information about the usefulness of ion exchange on the Miex-DOC resin in removing organic and non-organic substrates was the main reason for conducting this research.

Because of this, a study was conducted to determine the changes in water biostability level in water for human consumption with the use of pre-treatment using Miex-DOC resin.

MATERIALS AND METHODS

Research samples were collected from three surface waters (SW1, SW2 and SW3) and one infiltration water (IW) taken from a water treatment plant before treating it. Samples from each of the sources were taken six times (from April to August) and transported to the laboratory in thermostatic conditions (20 °C). Each sample had a volume of 15 L to determine the optimal conditions for

substrate removal. Samples were transported to the laboratory within 2 hours.

Water from these sources was pre-treated with use of Miex-DOC resin in laboratory conditions, in order to determine the changes in substrate levels in this process. In consequence, the possibility of limiting regrowth of heterotrophic micro-organisms with this process was evaluated.

Tests of changes in water biostability due to the ion exchange were conducted with macroporous Miex-DOC resin in container tests with full mixing. The samples (1 L per dose and contact time) were mixed at a speed of 150 rev/min, which, according to Molczan (2012), is optimal for removing organic substances.

During the tests, resin doses of 5, 10, 15, 20, 25 and 30 mL/L were used, with contact times of 30 minutes. Tests to determine the influence of resin-water contact time were conducted at a fixed resin dose of 20 mL/L with contact times of 10, 20, 30, 40, 50 and 60 minutes. For each test, fresh resin was used, which enabled a comparison of substrate elimination from different water sources.

For all samples of raw water and water after the ion-exchange process, the dissolved organic carbon (DOC) content, BDOC content and concentrations of non-organic substrates PO_4^{3-} , NO_3^- , NO_2^- and NH_4^+ were determined.

The DOC content was determined using a Hach-Lange total organic carbon analyzer. On the other hand, the BDOC content was determined to be the difference in DOC content before and after incubation with micro-organisms typical of the water environment. Details of the procedure are described in *Standard Methods* and Servais *et al.* (1989). The non-organic substrate content was determined using calorimetric methods, using a Shimadzu spectrophotometer, according to the American Public Health Association. For determining NH_4^+ concentration, the phenate method was used, while for NO_3^- the salicylic acid method was utilized. The phosphate concentration was analyzed according to the ascorbic acid method.

The following values from literature (Percival *et al.* 1998; LeChevallier 1999; Niquette *et al.* 2001) were used as limit values for water stability: BDOC = 0.25 gC/m³, N ($\text{NO}_3^- + \text{NO}_2^- + \text{NH}_4^+$) = 0.2 gN/m³ and PO_4^{3-} = 0.03 g PO_4^{3-} /m³.

Owing to trace concentrations of NO_2^- ions in all raw waters, their content was not taken into effect in evaluating water biostability, and was assumed to be equal to zero. The limit of detection for NO_2^- is 0.001 gN/m^3 . In addition, NH_4^+ ion content for pre-processed water was taken to be equal to that of raw water, as changes in ammonia ion content never exceeded the measurement error (0.005 gN/m^3).

Efficiency removal of substrate was calculated as $(C_o - C_e) \times 100/C_o$; %

where: C_o , C_e = substrate concentration before and after ion exchange, respectively.

RESULTS AND DISCUSSION

Source water characterization

The surface and infiltration water sources used in this study contained significant amounts of organic and non-organic substrates (Table 1). In all SW samples, the concentrations of substrates were found to be higher than

the limit values for water biostability. Only 33.3% of infiltration water samples, which were characterized by the lowest contamination levels, had a BDOC content smaller than 0.25 gC/m^3 .

SW sources were characterized by a high variability in substrate content, especially organic substances. Among non-organic nitrogen forms in these sources, nitrates dominated, constituting on average 87.6%, 91.1% and 88.7% of non-organic nitrogen for SW1, SW2 and SW3, respectively. On the other hand, this percentage for infiltration water was 73.6%. Infiltration water was also characterized by a much smaller variability in BDOC content.

Organic substrate removal

The organic substance removal effectiveness for different water sources ranged widely and depended on the resin dosage and the resin-water contact time (Table 2).

The organic substance (DOC) removal effectiveness values (up to 86.2%) agree with those found in other studies

Table 1 | Ranges of selected water quality indicators before and after ion exchange

Unit		Before ion exchange				After exchange ^a			
		SW1	SW2	SW3	IW	SW1	SW2	SW3	IW
DOC	gC/m^3	4.67–6.43	3.75–9.55	3.27–9.54	2.49–3.45	1.55–5.13	0.60–3.48	0.66–2.99	0.58–2.37
BDOC	gC/m^3	0.29–0.79	0.33–0.85	0.35–0.53	0.11–0.30	0.19–0.65	0.21–0.80	0.21–0.48	0.09–0.28
N- NO_3^-	gN/m^3	1.40–2.56	1.26–4.70	0.26–1.83	0.26–0.78	0.01–0.98	0.10–2.65	0.04–0.57	0.04–0.35
PO_4^{3-}	$\text{gPO}_4^{3-}/\text{m}^3$	0.28–0.56	0.06–0.22	0.05–0.28	0.15–0.24	0.02–0.24	0.01–0.14	0.00–0.14	0.01–0.18
N- NO_2^-	gN/m^3	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
N- NH_4^+	gN/m^3	0.05–0.28	0.02–0.33	0.09–0.45	0.13–0.38	0.05–0.28	0.02–0.33	0.09–0.45	0.13–0.38

^aThe range embraces values obtained from both research of the influence of the contact time and doses of resin on removal effectiveness. SW, surface water; IW, infiltration water.

Table 2 | Ranges of substrate removal effectiveness

	DOC_D	DOC_T	BDOC_D	BDOC_T	NO_{3D}^-	NO_{3T}^-	PO_{4D}^{3-}	PO_{4T}^{3-}
SW1	36.1–49.8	9.0–58.6	3.4–45.6	0.0–40.5	60.8–93.0	75.7–99.6	52.0–90.0	82.1–92.9
SW2	53.0–84.0	44.8–84.0	5.9–59.3	9.1–52.9	50.0–95.1	43.6–93.6	16.7–95.4	16.7–95.4
SW3	38.2–86.4	19.9–86.3	5.7–46.0	12.0–58.0	23.1–94.5	26.9–94.2	60.0–100.0	33.3–100.0
IW	40.0–80.8	17.7–75.2	3.4–43.3	6.0–30.0	61.5–93.6	42.3–88.3	50.0–95.8	21.7–95.8

D, removal effectiveness with varying resin dosages; T, removal effectiveness with varying water-resin contact times.

(Drikas *et al.* 2002; Mergen *et al.* 2008; Zhi-gang *et al.* 2010; Karpińska *et al.* 2013). However, despite the large effectiveness found for the Miex-DOC resin (Kaewsuk & Seo 2011; Shuang *et al.* 2014) in removing organic substances, removal effectiveness for BDOC was much smaller than that obtained for DOC and ranged from 3.4 to 59.3%. The efficiency was insufficient to obtain biostability for all samples, after 30 minutes contact time and 20 mL/L dose, with respect to BDOC (Table 1).

The amount of the removed biodegradable fraction of organic substances, irrespective of water type, was almost exactly proportional to its content in raw water (Figure 1). These results agree with those obtained in studies by Ding *et al.* (2012), who also found an almost proportional relationship between the initial organic substance concentration and its removal effectiveness during ion exchange using Miex-DOC resin.

Owing to low BDOC concentrations in raw water, the reduction in biodegradable organic substance concentration was also small and was in the range of 0.00–0.36 gC/m³, 0.03–0.45 gC/m³, 0.02–0.29 gC/m³ and 0.01–0.13 gC/m³ for SW1, SW2, SW3 and IW, respectively. Owing to this, achieving concentrations sufficient for biostability is difficult.

For SW1, none of the used resin dosages achieved a final BDOC concentration lower than 0.25 gC/m³. Only samples with a 40-minute contact time with a dosage of 20 mL/L achieved biostability with respect to BDOC content. However, this was only possible due to low biodegradable substance content in the raw water in this sample (0.29 gC/m³) (Figure 2(a) and 2(b)).

Similarly for SW3, ion exchange allowed for a BDOC reduction to biostability levels only in the case of low BDOC concentrations in raw water.

On the other hand, for SW2, a BDOC concentration lower than the limit concentration for biostability was found with a 40-minute contact time and a dosage of 20 mL/L.

After ion exchange ($D = 15$ mL/L) all infiltration water samples showed biostability with respect to organic substrate content, which was due to small concentrations of this organic fraction in all raw water samples and a small variability in BDOC concentrations (Table 1, Figure 3(a) and 3(b)).

Differences in the removal effectiveness for the different water sources may be explained by different types of organic substances that comprise the biodegradable fraction. Kaewsuk & Seo (2011) have shown that simple carboxyl acids,

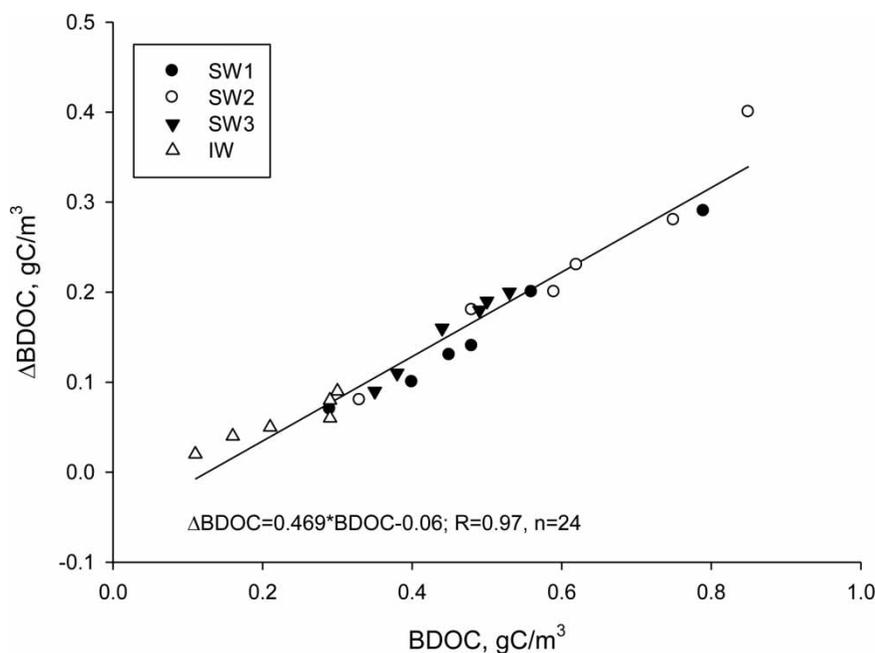


Figure 1 | Relationship between biodegradable substance concentration in raw water and the degree of concentration reduction (dose 20 mL/L and contact time 30 minutes).

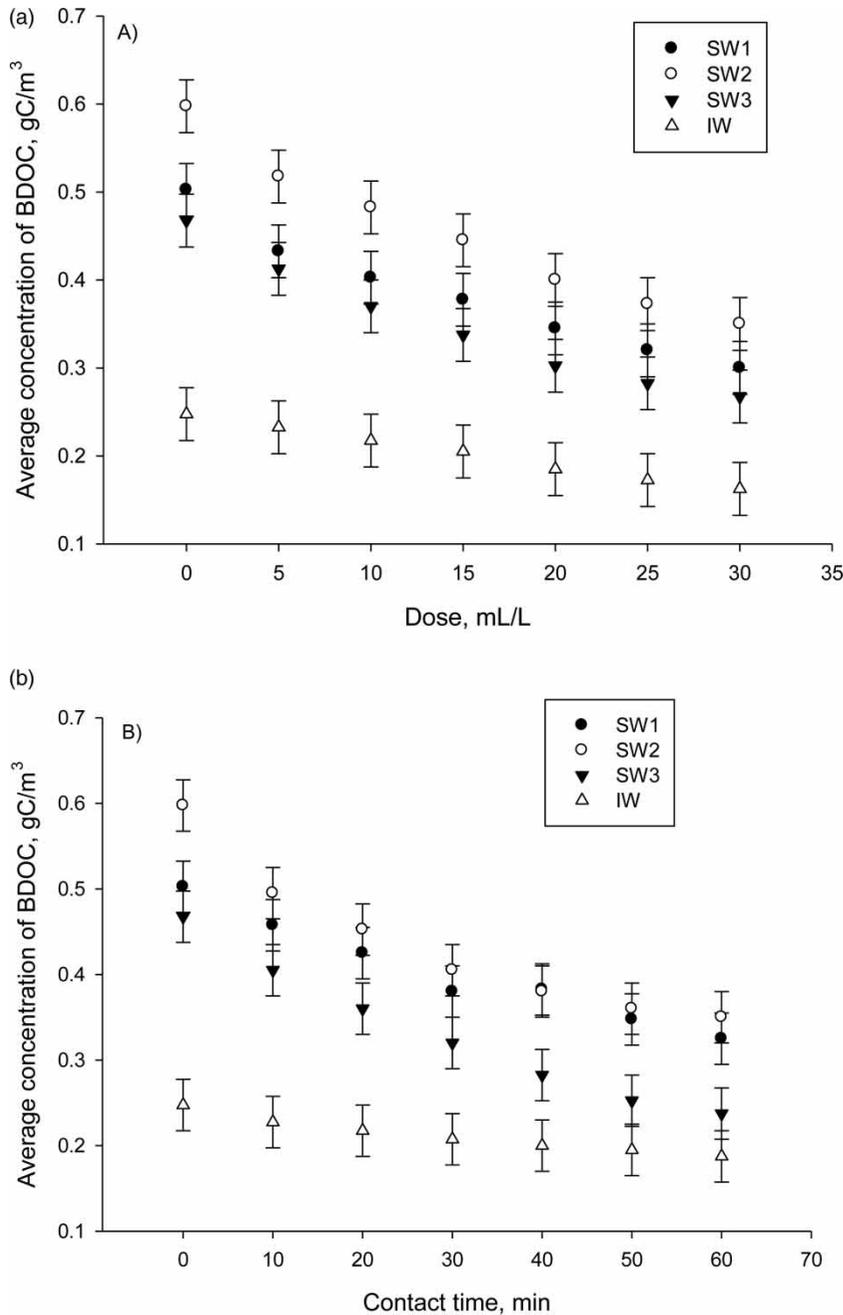


Figure 2 | BDOC removal (a) with varying resin concentration and (b) with varying contact time.

which are part of the biodegradable fraction, are affected only slightly by ion exchange with the Miex-DOC resin.

The BDOC removal effectiveness that was obtained during this study is sufficient for ensuring biostability only in the case of lowly contaminated infiltration water or in periods of low SW contamination. Owing

to this, with higher contamination levels, ion exchange with the Miex-DOC resin will not ensure sufficient biodegradable substance removal. In such cases, it is necessary to eliminate these substrates with other unit water treatment processes, e.g. biofiltration (Yu *et al.* 2002).

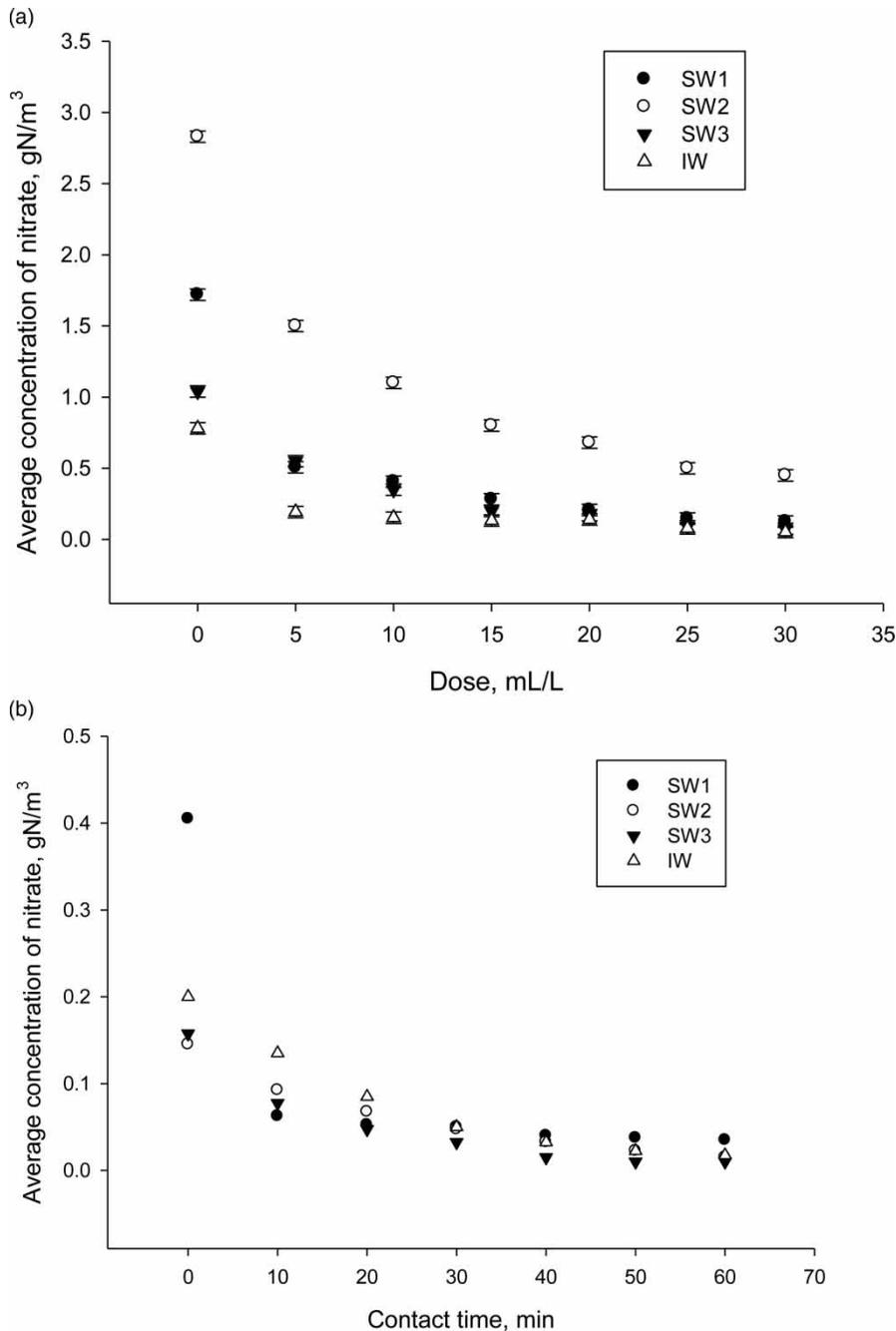


Figure 3 | Nitrate removal (a) with varying resin concentration and (b) with varying contact time.

BDOC removal effectiveness may be approximated by the Freundlich isotherm. A similar finding has been found for studies concerning geosmin removal (Drikas *et al.* 2009) and other micro contaminants (Liu *et al.* 2011). It means that most of the BDOC was removed during the adsorption process and not during ion

exchange. The Freundlich isotherm represents a simplified adsorption isotherm.

However, due to the danger associated with an excessively high content of biodegradable substance, the Miex-DOC process should be augmented by other unit treatment processes.

Achieving effective BDOC elimination in water pre-treatment during an ion-exchange process with Miex-DOC is even more crucial due to the fact that conventional water treatment systems have a low effectiveness in removing BDOC and do not achieve water biostability. Only the biofiltration process is capable of increasing the biodegradation effectiveness of this organic fraction; however its effectiveness is frequently limited by other factors (Jianga *et al.* 2011).

Nitrate removal

Nitrate ion removal in conventional water treatment systems is very difficult and requires, for example, the use of biological processes (Wolska 2014), which most often are not sufficiently effective in removing nitrates from water.

The results of the ion-exchange process using the Miex-DOC resin show a very high effectiveness in removing nitrate ions (Table 2, Figure 3(a) and 3(b)). An efficiency of nitrate removal greater than 75% was found for 89.9% of samples for SW1, 75.0% for SW2, 63.9% for SW3 and 72.2% for IW.

Relative to nitrate removal, contrary to what was found with BDOC, there was no unambiguous relationship between nitrate removal effectiveness and content in raw water. The mean NO_3^- removal effectiveness for each water source was as follows: SW1 > SW2 > IW > SW3. Despite the large nitrate removal effectiveness, their concentrations in treated water were not sufficiently low to ensure water biostability in 39.8% of samples (Figure 3(a)).

All samples of infiltration water were characterized by nitrate concentrations lower than the limit value with respect to water biostability at resin dosages of just 10 mL/L. For SW3 this dosage amounted to 20 mL/L, and for SW1 even 30 mL/L. On the other hand, for 25% of water samples from SW2, irrespective of resin dosage, the nitrate content was higher than 0.2 gN/m^3 . Even increasing the resin contact time at a dosage of 20 mL/L did not achieve a sufficient elimination of these anions from SW2 and SW1.

The most effective contact time was 30 minutes for all types of water (Figure 3(b)), as longer contact times result in only small improvements in NO_3^- removal.

In the majority of water samples (61.1%), the NO_3^- concentration after ion exchange was lower than the limit value for biostability. Only for very large nitrate ion

concentrations (4.6 gN/m^3) was it not possible to achieve satisfactory process effectiveness. The lower nitrate ion removal effectiveness obtained by Tang *et al.* (2013) is due to lower concentrations in the studied waters.

The effectiveness that has been obtained shows the utility of ion exchange using Miex-DOC resin in achieving water biostability. Such a large nitrate removal effectiveness cannot be provided by any of the conventional water treatment processes, and frequently the effectiveness of biofiltration is limited by too low phosphate concentrations in input water (Jianga *et al.* 2011).

Phosphate removal

Phosphate ion removal effectiveness, as was the case for nitrates, was very high irrespective of water type (Table 2). Generally, the reduction in these substrate concentrations was larger for higher raw water phosphate contamination levels (Figure 4).

Consequently, achieving phosphate concentration sufficient for biostability was possible for all water from SW2, SW3 and IW with a resin dosage of 20 mL/L. To obtain such a concentration in 50% of samples from SW1 it was necessary to increase resin dosages to 30 mL/L, and with the remainder of samples from this source, biostability was not achieved within the range of dosages and contact times used in this study. With respect to this water source, even increasing the contact time to 60 minutes with a resin dosage of 20 mL/L did not allow biostability to be achieved in 50% of samples (Figure 5(a) and 5(b)).

In the majority of water samples, a dosage of 20 mL/L and a contact time of 30 minutes were sufficient to achieve biostability with respect to phosphate concentrations. However, such effective phosphate removal in the pre-treatment stage is not necessary, as the ions are effectively eliminated during the coagulation process with sedimentation or during filtration (Lethola *et al.* 2002).

The results of this study show a higher effectiveness in removing non-organic substances than organic substances, and in the majority of cases, a higher effectiveness in removing nitrates than phosphates (57.1% of samples). This is probably because unlike in Tang's study, the water sources used in this study had higher NO_3^- concentrations than PO_4^{3-} concentrations. The average removal effectiveness

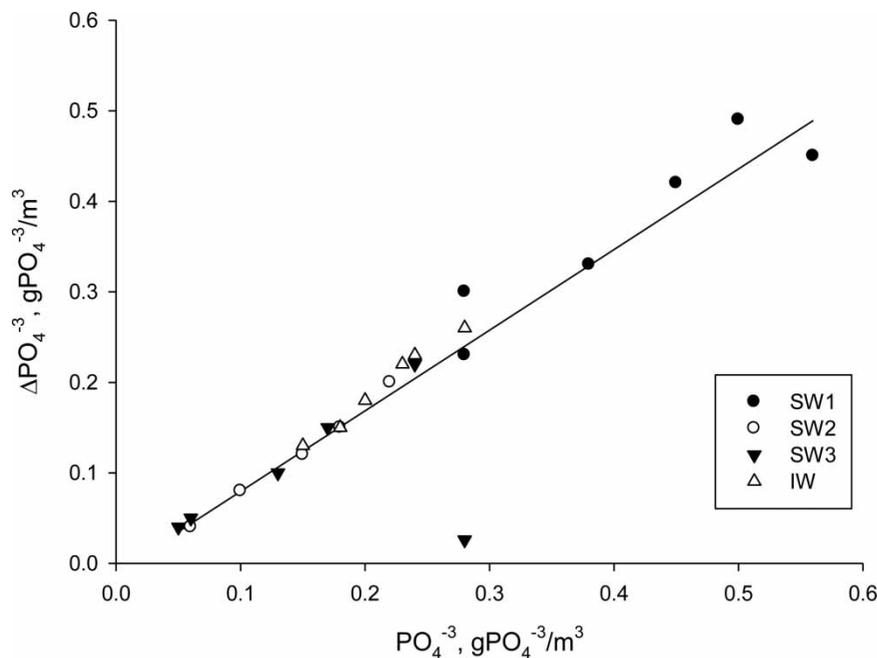


Figure 4 | Relationship between raw water phosphate content and removal effectiveness (contact time 30 minutes; dose 20 mL/L).

was 84.4% for NO_3^- and 80.6% for PO_4^{3-} for all studied waters with a dose of 20 mL/L and contact time of 30 minutes. This hypothesis is supported by the fact that a higher phosphate removal effectiveness was found only for samples characterized by small NO_3^- concentration. Consequently, this was the case only for infiltration water. A general increasing trend was observed between NO_3^- concentrations and it decreased during the ion-exchange process.

The effectiveness of phosphate ion removal depends on dose and contact time with resin (Figure 5(a) and 5(b)). The majority of removal was in the first 30 minutes of the process and in doses of 5, 10, 15 and 20 mL/L.

CONCLUSIONS

- The biodegradable organic substance fraction is much less susceptible to ion exchange than DOC.
- The degree of BDOC elimination was sufficient for ensuring water biostability only in water samples that had a low level of BDOC contamination.
- Irrespective of water type, a very effective elimination of non-organic substrates was found, and therefore these

substrates may be the limiting factor in micro-organism growth in the water distribution system.

- The use of Miex-DOC resin in water pre-treatment allows for a significant limiting of the problem of a lack of biostability of water introduced into the distribution system, especially with respect to nitrate ion content.
- The effectiveness of phosphate removal for SW sources found in this study was lower than that found for nitrates. However, for infiltration water, which has lower nitrate content, the opposite was found.
- The effectiveness of removing both organic substrates and phosphate depended on, above all, raw water contamination levels.
- An increase in resin dosages in the range of 5–20 mL/L yielded a very large increase in substrate elimination effectiveness. However, further increases in resin dosage only slightly increased removal effectiveness.
- Significant increases in process effectiveness were found for contact times up to 30 minutes. Further increases in contact time increased substrate removal effectiveness only slightly.
- Optimal parameters of ion-exchange processes should be selected on the basis of substrate concentrations in raw water.

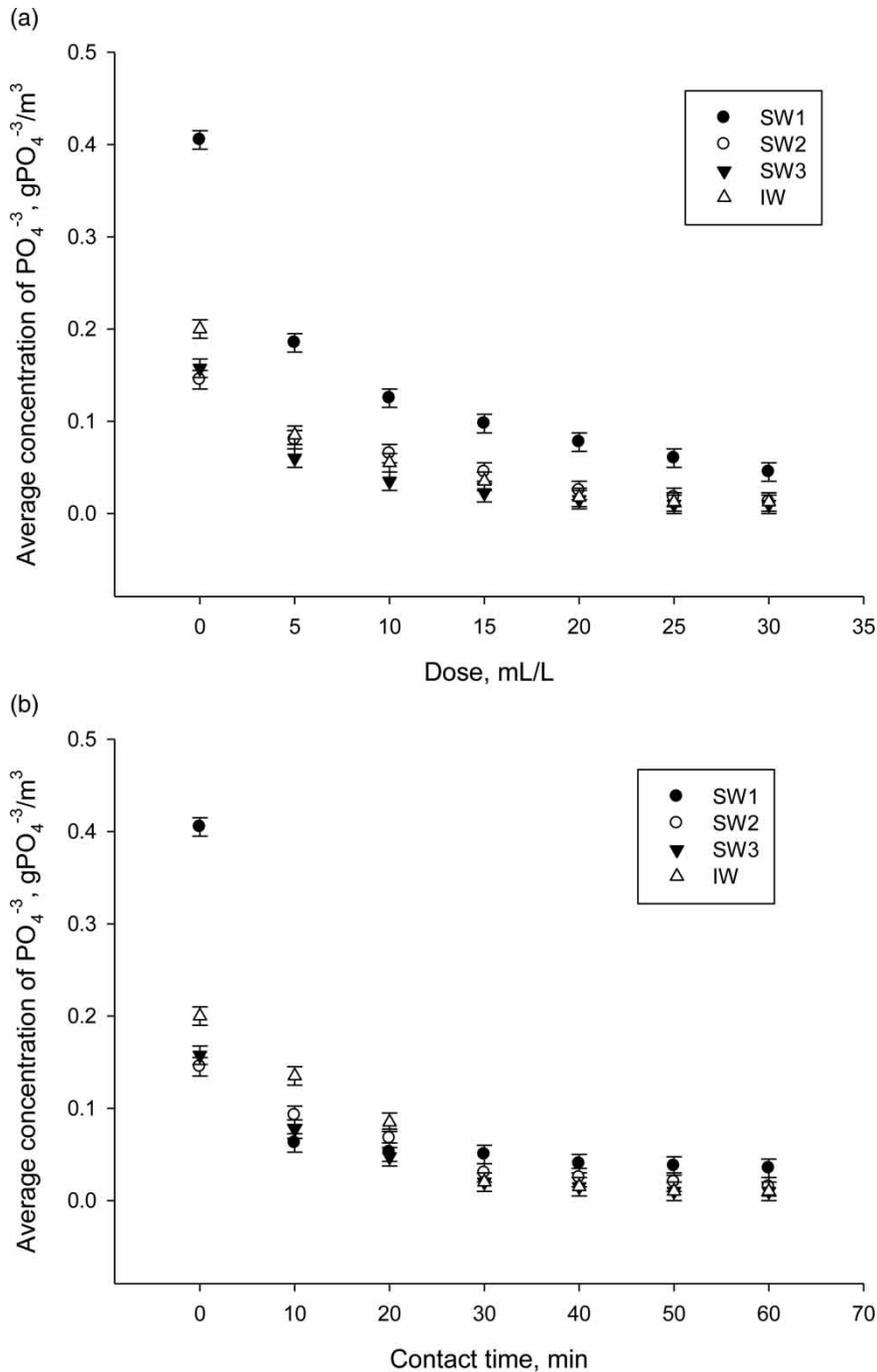


Figure 5 | Phosphate removal (a) with varying resin concentration and (b) with varying contact time.

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