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# INFLUENCE OF $\text{Ca}(\text{OH})_2$ ON THE EXTRACTION EFFICIENCY OF NITROAROMATIC COMPOUNDS FROM SOILS

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High concentrations of TNT and related nitroaromatic compounds (NAC) can still be found in the soils. To evaluate the alkaline hydrolysis of NAC as a new remediation technology, two highly contaminated soils were treated under alkaline conditions using  $\text{Ca}(\text{OH})_2$  as base. However, instead of the expected decrease, a temporary or even permanent increase was observed for several NAC. The extent of the intensity was affected by  $\text{Ca}(\text{OH})_2$  concentration, suggesting the existence of desorption processes in the soil. The extent of the increase also depends on the soil investigated, the highest being observed with 1,3,5-trinitrobenzene ( $239 \text{ mg kg}^{-1}$  compared to the baseline concentration of  $24 \text{ mg kg}^{-1}$ ) in the HTNT2 soil. This indicates incomplete NAC extraction and, hence, too low NAC concentrations measured in soils when conventional extraction procedures are used.

*Keywords:* Explosives; Nitroaromatic compounds (NAC); Desorption; Extraction efficiency; Accuracy of data

## INTRODUCTION

Nitroaromatic compounds (NAC) are widely used chemicals. In particular the widespread use of explosives such as 2,4,6-trinitrotoluene (TNT) presents a serious and potentially hazardous contamination problem. Disposal of TNT and related compounds during production, handling, and storage has frequently led to soil and subsurface contamination [1,2]. Many of these compounds pose a significant toxicological and ecotoxicological threat [3–7]. Thorough risk assessment and the evaluation of appropriate remediation strategies of such contaminated sites require reliable analytical methods, since the resulting consequences depend on the values measured. However, trace analysis of organic compounds is difficult due to the complexity and diversity of soil matrices.

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Sorption of NAC to soil constituents is influenced by both substance and soil-related properties. Adsorption is frequently associated with soil organic matter. Hydrophobic distribution of organic compounds into organic matter is commonly assumed to be the major sorption mechanism, especially in soils with a high organic content [8]. However, Pennington and Patrick [9] found that adsorption isotherms of TNT are most closely correlated with extractable iron, cation exchange capacity, and clay percentage, even in the case of soils with high organic content. Haderlein *et al.* [10,11] demonstrated a specific adsorption of NAC to clay minerals. Weissmahr *et al.* [12] suggest an electron donor–acceptor (EDA) complex between oxygens of the siloxane surface of the clays and the NAC.

Furthermore, sorption of organic compounds on soils appears to be affected by the time over which the compounds have been in contact with the soils [13]. In addition, increasing ageing of soils is frequently accompanied by decreasing extractability of the NAC. This may be due to changes of binding properties, sorption processes and diffusion or migration of compounds into soil particles [14].

To obtain basic data for alkaline hydrolysis of NAC as a novel remediation technology of contaminated soils, two soils from two former ammunition plants were treated at pH 11 and 12 using  $\text{Ca}(\text{OH})_2$  as base. Good hydrolytic rates were achieved for most of the higher concentrated compounds [15,16]. However, for several other NAC a temporary or even lasting increase of the amounts was noted instead of the expected decrease. The data presented here indicate that an incomplete extraction of NAC from soils takes place when common extraction procedures are used. Thus, the concentrations measured in the soils may be too low, which may have implications in risk assessment studies.

## EXPERIMENTAL

### Alkaline Treatment of Soil

Soil samples were collected from the former ammunition plant Hallschlag (soil HTNT2) and the burning place of the former ammunition plant Torgau/Elsnig (soil ELBP2), both located in Germany. Detailed information of physical, chemical, biological and ecotoxicological soil characteristics are described elsewhere (Eisenträger *et al.* [17] for HTNT2 and Müller *et al.* [18] for soil ELBP2).

Prior to treatment, the soils were passed through a 2 mm sieve and air-dried. To achieve alkaline hydrolysis, 5 g of the soil samples were transferred into reaction vessels (100 mL Erlenmeyer) and then stirred with 50 mL of a  $\text{Ca}(\text{OH})_2$  solution at pH 11 and 12, respectively. Regulation of the pH was performed by a programmable 8052 AH Basic microcontroller and was kept constant within a range of  $\pm 0.1$  pH units by controlled addition of a saturated and filtered  $\text{Ca}(\text{OH})_2$  solution. Alkaline treatment was carried out for a period of 14 days using HTNT2 soil at pH 11 and 12 and ELBP2 soil at pH 11 and for 7 days using ELBP2 soil at pH 12.

### Extraction of Samples

At predefined times the respective soil samples were neutralized with HCl to stop the reaction. Subsequent centrifugation was carried out at 2500 rpm for 10 min.

50 mL of the supernatant were used for triplicate extraction by shaking with 25 mL ethylacetate for 3 min each. The combined organic phases were dried with anhydrous sodium sulphate, evaporated to about 0.1 mL and redissolved with 1 mL methanol.

The remaining soil was dried with  $\text{Na}_2\text{SO}_4$  and extracted in a Soxhlet device with 80 mL *tert*-butylmethylether (SupraSolv, Merck) for 3 h. The extract was then dried over anhydrous sodium sulphate and evaporated to about 0.1 mL. The samples were redissolved with *tert*-butylmethylether.

### Gas Chromatography (GC)

NAC were analysed using a GC equipped with an ECD (Hewlett Packard 5890 series II) and a cold injection system KAS 3 (Gerstel, Mülheim a.d. Ruhr, Germany). Separations were performed on a HT8 column (SGE, Weiterstadt, Germany) using helium as the carrier gas. The following temperature program was used: 2 min at 40°C, 40–130°C with 10°C min<sup>-1</sup>, 1 min at 130°C and then 130–250°C with 3°C min<sup>-1</sup>.

### Evaluation of Results

The total amount of each compound in the reaction vessel was calculated by adding the amounts of the compound in both the liquid and the solid phases. These quantities relate to the 5 g of soil used in the experiments. Therefore they were multiplied with a factor of 200 to achieve the corresponding quantities for 1 kg soil (Table I). The amounts of the NAC measured in the untreated soil were used as reference values (100%) to calculate recovery rates.

TABLE I NAC detected in the untreated HTNT2 and ELBP2 soils, and maximum recovery rates ( $RR_{\max}$  as sum of solid and liquid phases) measured during alkaline soil treatment.

Substance	Untreated soil $C_0$ (mg/kg <sup>-1</sup> )	pH 12 $RR_{\max}$		pH 11 $RR_{\max}$	
		(mg)	(%)	(mg)	(%)
HTNT2					
1,3-DNB	8.7	17.0	(195)	26.4	(303)
TNB	24.2	88.6	(366)	238.6	(986)
3,5DN-An	2.8	12.0	(428)	22.6	(808)
2,3-DNT	2.8	5.0	(177)	5.2	(184)
2,4-DNT	289.2	cda		cda	
2,6-DNT	70.5	77.6	(110)	82.5	(117)
3,4-DNT	4.8	8.8	(183)	10.2	(212)
TNT	16109.8	cda		cda	
2A-4,6DNT	66.7	cda		cda	
4A-2,6DNT	80.3	cda		cda	
ELBP2					
1,3-DNB	0.07	3.6	(5206)	0.2	(277)
TNB	1.10	18.1	(1648)	4.8	(438)
3,5DN-An	0.08	7.0	(8756)	0.9	(1148)
2,3-DNT	0.19	3.0	(1561)	0.3	(134)
2,4-DNT	142.7	cda		cda	
2,6-DNT	58.0	85.3	(147)	65.5	(113)
3,4-DNT	0.13	2.7	(2045)	0.3	(197)
TNT	115.7	cda		141.2	(122)
2A-4,6DNT	0.69	5.3	(767)	1.8	(266)
4A-2,6DNT	0.51	16.3	(3201)	3.1	(617)

cda: continuously decreasing amounts due to alkaline hydrolysis

## RESULTS AND DISCUSSION

The concentrations of the NAC detected in the untreated soils are summarized in Table I. The major contaminants found in both soils were TNT and the dinitrotoluenes 2,4-DNT and 2,6-DNT. In addition, HTNT2 contained the two aminodinitrotoluenes 2A-4,6DNT and 4A-2,6DNT as well as 1,3,5-trinitrobenzene (TNB). Several related NAC were detected in lower concentrations.

The maximum amounts of NAC measured during alkaline treatment of HTNT2 and ELBP2 are also summarized in Table I. The values represent the maximum recovery rates in both the solid and the aqueous phases. Regarding HTNT2 the TNT concentration continuously declined by alkaline treatment at pH 11 and pH 12. Regarding ELBP2 an almost complete TNT hydrolysis was also achieved at both pH 11 and pH 12. As in the case of HTNT2 the concentration in the solid phase of ELBP2 continuously decreased. However, at pH 11 the concentration in the aqueous phase temporarily increased, even more than expected. During the first two days up to 122% of the initial TNT was detected in both the solid and the aqueous phases. Subsequently, TNT hydrolysis prevailed and TNT almost completely disappeared [15,16].

A similar increase in both the solid and the aqueous phases was observed for most of the NAC. The course of the 1,3-DNB recovery rates *versus* time is representative for many nitroaromatics (Fig. 1a for ELBP2 and Fig. 1b for HTNT2). Regarding ELBP2 at pH 12 a continuous increase of up to 5206% was measured. With the experimental conditions used 1,3-DNB slowly approached its peak after 7 days. An increase of 1,3-DNB was also observed at pH 11, although it reached a maximum amount of 277% 'only'. At pH 11 it slightly decreased after 4 days, whereas no hydrolysis was noted at pH 12. This may be due to the massive increase at pH 12, that may superimpose on a potential 1,3-DNB hydrolysis.

Regarding HTNT2 a higher maximum amount was reached at pH 11 (303%) compared to pH 12 (195%). However, during the first hours a more rapid increase was observed at pH 12 compared to pH 11, indicating that a greater increase of 1,3-DNB may have taken place at pH 12 compared to pH 11. This increase may be superimposed by alkaline 1,3-DNB hydrolysis, as suggested by the decreasing amounts after 1 day. Since its hydrolysis is more effective at pH 12, the sum of these two processes may lead to a lower total increase at pH 12 compared to pH 11.

Figure 2 shows the distribution of TNB between the solid and the aqueous phases during alkaline treatment with HTNT2. At pH 11 a steady increase in recoveries was noted, first in the solid phase, and later also in the aqueous phase, with maximum amounts reaching 500% in each case. Within the first 4 days this led to an increase of up to 986%, corresponding to  $239 \text{ mg kg}^{-1}$  TNB. This represents a ten-fold higher concentration measured in the treated HTNT2 soil compared with the initial value of  $24 \text{ mg kg}^{-1}$  TNB measured in the untreated soil. TNB hydrolysis may be only partial at pH 11. Hence, 272% of TNB was still detected at the end of the alkaline treatment.

Of the NAC present in the two soils only those compounds continuously decreased during alkaline treatment, where hydrolysis prevailed, namely TNT, 2A-4,6DNT, 4A-2,6DNT, and 2,4-DNT in HTNT2 soil and TNT (pH 12), and 2,4-DNT in ELBP2 soil [16]. The amounts of all other compounds temporarily or even persistently increased during alkaline treatment of HTNT2 (14 days for pH 11 and 12) and ELBP2

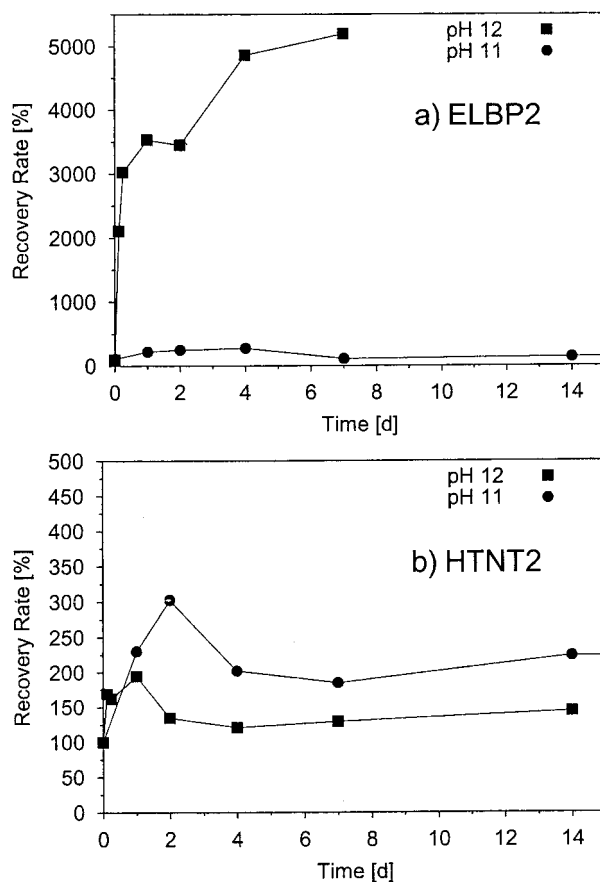


FIGURE 1 Total recovery rates (solid and liquid phases) of 1,3-DNB detected during alkaline treatment of ELBP2 (top) and HTNT2 (bottom) at pH 11 and 12.

(14 days for pH 11, 7 days for pH 12), as shown in Figs 1 and 2. Maximum recovery rates of up to 8756% (3,5DN-An) were observed for ELBP2 soil at pH 12 and up to 986% (TNB) for HTNT2 soil at pH 11. Regarding the HTNT2 soil, the increase of the amounts at pH 12 was generally less pronounced compared to pH 11, whereas in ELBP2 the increase was higher at pH 12 than at pH 11. This might be due to the significant difference between HTNT2 and ELBP2 regarding the distribution of the NAC among the soil and the aqueous phases during alkaline treatment [16].

The observed increase of the compounds during alkaline hydrolysis is not attributable to inhomogeneities of the HTNT2 and ELBP2 soil samples, since the soils were well homogenized prior to the investigations. In addition, the concentration of the NAC in the untreated soils was determined using three independent samples. With respect to the high TNT concentration, each sample of untreated HTNT2 soil was even extracted three times to make sure that complete extraction of the NAC was achieved. Furthermore, the increase of substances like 1,3-DNB and TNB and their subsequent decrease developed rather continuously and was too pronounced to be explained by analytical errors only. In addition, the observed increase of certain compounds during alkaline hydrolysis is not attributable to the formation of these compounds as

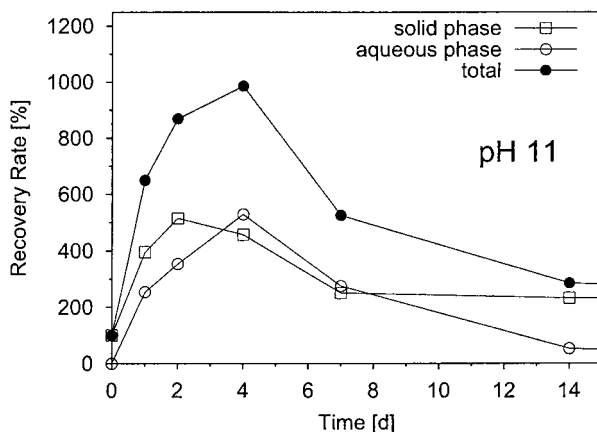


FIGURE 2 TNB recovery rates in the solid and liquid phases, and total recovery rates measured during alkaline treatment of HTNT2.

byproducts of alkaline TNT hydrolysis. In order to form TNT byproducts such as 1,3-DNB or TNB, a nitro group and/or the methyl group of TNT must be split off, followed by hydrogenation of the remaining molecule. Regarding ELBP2 soil and pH 12, 18% of the TNT must be hydrolysed in this way to achieve the temporary TNB increase of up to 1648%. Formation of TNB was not observed during homogeneous alkaline TNT hydrolysis at pH 12 [15]. In addition, these kinds of reactions have never been described so far. Also, this would not explain the increase of TNT itself of up to 122% observed during alkaline treatment of ELBP2 at pH 11. Hence, the compounds certainly do not represent byproducts of alkaline TNT hydrolysis.

Instead, this sometimes very pronounced increase during alkaline treatment and the differences in the increase at pH 11 and 12 suggests the existence of desorption processes of the compounds from the soil. Several alternative hypotheses are possible to interpret the observations. The observed increase of the NAC may be due to pH effects. At pH 11 or 12, disaggregation of clays and other soil aggregates occurs. In addition, soil organic matter becomes more soluble at higher pH values since dissociation increases the negative surface charge of the organic particles or molecules. Intermolecular and intramolecular electrostatic repulsion is believed to be responsible for this increased solubility. In the "random coil" model of humic substances, the repulsive forces cause an unfolding and expansion associated with the dispersion and dissolution of the organics [8]. Furthermore, the addition of  $\text{Ca}^{2+}$  may change the soil aggregate structure to particles with increased permeability and drainage of the soil [19]. Haderlein *et al.* [10,11] and Weissmahr *et al.* [20] demonstrated a specific adsorption of NAC to clay minerals which strongly depends on the cation composition in the soil solution. The authors showed a significant influence of  $\text{Ca}^{2+}$  on the adsorption and desorption of NAC.

Our investigations indicate that the addition of  $\text{Ca}(\text{OH})_2$  for alkaline hydrolysis may induce desorption of the NAC from the soil. This in turn leads to better NAC extractability by the solvent used for extraction. However, it is impossible to identify the underlying desorption processes on the basis of the data presented here. All mechanisms described above may contribute to the NAC desorption observed.

Although the underlying processes remain unclear, the increase in NACs demonstrates that without the desorption processes observed during alkaline treatment only partial NAC extraction may be achieved by means of common extraction procedures. To estimate the true baseline NAC concentrations in the untreated soils, two situations associated with alkaline treatment must be distinguished: a situation where desorption occurs without hydrolysis and a situation where desorption and hydrolysis occur simultaneously. With ELPB2 soil at pH 11 the NAC amounts continuously increased except for 2,6-DNT. No NAC decrease was noted during alkaline hydrolysis, indicating that only desorption processes occurred. The same applies to 1,3-DNB, 2A-4,6DNT and 4A-2,6DNT at pH 12. In this case the maximum amounts listed in Table I may reflect the true baseline amount of NAC in the original untreated ELPB2 soil. Regarding all NAC contained in HTNT2 soil at both pH values an increase was observed, followed by a decrease. The same is true for TNB, 3,5DN-An, 2,3-DNT, 2,4-DNT and 3,4-DNT in ELBP2 at pH 12. The increase followed by a decrease suggests that desorption and hydrolysis occurred simultaneously during alkaline treatment. In this case, the baseline NAC concentrations in the untreated soils must be even higher than the maximum values listed in Table I.

For the determination of NAC in soil matrices, extraction is commonly performed using an ultrasonic bath with methanol [21], acetone [22], or acetonitrile [23] or a Soxhlet device in combination with diethylether [24]. In our experiments the soils were extracted with a Soxhlet device and *tert*-butylmethylether. This method yielded results similar to those obtained with other frequently used methods, e.g., the Soxhlet extraction of Holland and Holighaus [24]. Organic solvents mainly disrupt non-polar interactions between the soil matrix and the NAC. In this case electrostatic forces such as the specific EDA complex interaction between the clays and the NAC may become predominant and the extraction achieved with common procedures may be incomplete. The observed increase of several NAC during alkaline treatment of the HTNT2 and ELBP2 soils indicates that the addition of  $\text{Ca}(\text{OH})_2$  may disrupt electrostatic forces. As a consequence, the non-extracted share of NAC becomes accessible to the extraction procedure.

Accuracy is a key attribute of data quality. Accuracy refers to the closeness of the results between the measured and the actual (true) analyte concentration in the sample. Analytical methods yield reproducible results with adequate inter-laboratory comparability when using reference materials or fortified real field samples along with specified extraction procedures. The data presented here suggest that extraction of NAC from soils may be incomplete and that little may be known about extraction efficiency and the true analyte concentration of NAC in soils. However, generalization of the observed phenomenon may be problematic since extraction efficiency depends not only on the chemical properties of the compounds and the extraction procedure used but also on soil solution and soil-related properties.

In addition, the extractability of NAC frequently diminishes with the "ageing" of soils when conventional extraction procedures are used. This may be due to changes of binding properties, sorption processes and diffusion or migration of compounds into soil particles [14]. Due to soil ageing over long periods, adsorption of nonextractable NAC is frequently considered an irreversible process. However, our results suggest that desorption and remobilization of NAC may be influenced by the  $\text{Ca}^{2+}$  concentration in the soil solution, with higher concentrations leading to desorption and thus to remobilization of NAC in soils. This may become important especially



on former ammunition plants where plenty of rubble resulting from accidents, bombardments, and war-related destruction of manufacturing buildings can still be found. Since these materials contain mortar and lime with a high  $\text{Ca}^{2+}$  ion content leaking water may lead to increased  $\text{Ca}^{2+}$  concentrations in soil solutions and thus to the observed desorption effects.

Since risk assessment of remediation strategies of contaminated soils require accurate data, reliable extraction procedures of NAC in soils are essential. Hence, analytical methods should be evaluated with respect to extraction efficiency and the different binding forces of NAC on soils in combination with real field soils.

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