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A macromolecular *N*,*N*-dichlorosulfonamide as oxidant for cyanides

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Abstract

The possibility of the use of a redox copolymer—a macroporous poly(styrene-co-divinylbenzene) resin having *N*,*N*-dichlorosulfonamide groups—as macromolecular oxidant for destruction of cyanide ions contained in dilute aqueous solutions has been investigated in this report. The resin contains two atoms of active chlorine in functional groups and shows strong oxidising properties. It was employed in static and flow systems for transformation of cyanide ions to non-toxic compounds. The reactions were carried out in aqueous media containing 26–2600 mg CN^-/dm^3 at pH in the range 10–14. The data showed that the macromolecular oxidant easily decomposes cyanides forming cyanates which are unstable in aqueous solutions—they hydrolyse to carbonates and ammonia. Both of the two atoms of active chlorine attached to the sulfonamide group of the reactive copolymer show oxidising properties in relation to cyanide ions but their activity is different in examined conditions. In strong alkaline medium one of these atoms easily quits the resin phase and instantly oxidises cyanides, whereas the second of them bound more strongly oxidises cyanide in static condition in a longer time. To utilise with good efficiency both of the two atoms of active chlorine for destruction of cyanide ions in processes carried out in flow systems it is necessary to maintain the level of basicity of the influent as low as possible.

1. Introduction

This work is a continuation of the research on destruction of cyanide ions using reactive polymers. In a previous paper the studies on a new way of decomposition of cyanide ion in dilute aqueous solutions were reported [1]. This way depends on the action of a redox copolymer—a macromolecular oxidising agent containing active chlorine. This agent has a macroporous poly(styrene-co-divinylbenzene) matrix structure and *N*-chlorosulfonamide functional groups. Owing to the presence of chlorine atoms with oxidation number +1 and a very high concentration of active chlorine in the resin bed (1.35 mol of active chlorine/dm^3) this copolymer was useful to destruction of cyanide ions in static and dynamic conditions. Using this copolymer in column systems it was possible to obtain effluents free of cyanides (final concentration of CN^- ion below 0.05 mg/dm^3). Cyanide ions oxidised by this redox copolymer transform to cyanate which are far less toxic than cyanides. Cyanate ions are unstable in aqueous solutions—they hydrolyse to carbonate and ammonia.

The present study aims at the examination of the possible use of a redox copolymer containing $-SO_2NCl_2$ functional groups for destruction of cyanide ion in dilute aqueous solutions according to the equation:

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$$[P]\text{–}SO_2NCl_2 + 2CN^- + 2H_2O \rightarrow [P]\text{-}SO_2NH_2 + 2OCN^- + 2HCl \quad (1)$$

[P] means S/DVB

This copolymer is a macromolecular *N*,*N*-dichlorosulfonamide resin (abbreviated as DCSR) comprising a macroporous S/DVB copolymer bearing $–SO_2NCl_2$ functional groups. It contains two chlorine atoms with the oxidation number +1 and like the *N*-chlorosulfonamide resin shows oxidative properties [2–4]. It oxidises several organic and inorganic compounds (sulphite and thiosulphate ions to sulphate; thiocyanate ion to sulphate, carbonate and ammonia; hydrazine to nitrogen). Moreover, it may be used for chlorination of organic compounds (phenol to *p*-chlorophenol) and for water disinfection.

The purpose of the present work was to examine the oxidising properties of DCSR in relation to cyanide ions contained in aqueous media for transformation them to less toxic products. It was interesting to compare the reactivity of *N*,*N*-dichlorosulfonamide groups with *N*-chlorosulfonamide groups both attached to the same macromolecular support in similar reaction conditions. One should remember carrying out the experiments with DCSR and cyanide solutions—that on the one hand cyanide solutions must show alkaline reaction (hydrogen cyanide—one of the most poisonous substances known—can readily be displaced from the solution of its alkali salts at pH below 9) and on the other hand DCSR may not be sufficiently stable in examined alkaline media. Earlier experiments concerning the stability of DCSR in aqueous media showed its highest stability in neutral and acidic medium even at elevated temperature. In alkaline medium a sample of DCSR after 20 days of contact with 1 M NaOH in static condition at room temperature showed an active chlorine content of 69.3% (in reference to the starting value) [4].

In the chemistry of low molecular organic compounds, it is well known that the low molecular compounds containing $–SO_2NCl_2$ groups—*N*,*N*-dichlorobenzenesulfonamide (Dichloramine-B) and *N*,*N*-dichloro-*p*-toluenesulfonamide (Dichloramine-T) (an analogue to DCSR) transform to *N*-monochloroderivatives by the action of aqueous sodium hydroxide solutions:

$$p\text{-}CH_3\text{–}C_6H_4\text{–}SO_2NCl_2 + 2NaOH \rightarrow p\text{-}CH_3\text{–}C_6H_4\text{–}SO_2NClNa + NaOCl + H_2O \quad (2)$$

The reaction carried out in strong conditions—with 10% NaOH at 80°C—is one of the methods of synthesis of low molecular *N*-chlorosulfonamides starting from *N*,*N*-dichloroderivatives.

Dichloramine-B and Dichloramine-T are substances with known oxidative properties. They found an analytical application e.g. for titrimetric determination of cyanide ion in aqueous media [5–7]:

$$p\text{-}CH_3\text{–}C_6H_4\text{–}SO_2NCl_2 + 2CN^- + 2H_2O \rightarrow p\text{-}CH_3\text{–}C_6H_4\text{–}SO_2NH_2 + 2OCN^- + 2HCl \quad (3)$$

2. Experimental

2.1. Resin

The copolymer having *N*,*N*-dichlorosulfonamide groups was prepared in the following way:

$$[P]\text{-}SO_3H + PCl_5 \rightarrow [P]\text{-}SO_2Cl + POCl_3 + HCl \quad (4)$$

$$[P]\text{-}SO_2Cl + 2NH_3(aq). \rightarrow [P]\text{-}SO_2NH_2 + NH_4Cl \quad (5)$$

$$[P]\text{-}SO_2NH_2 + NaOCl \rightarrow [P]\text{-}SO_2NClNa + H_2O \quad (6)$$

$$[P]\text{-}SO_2NClNa + NaOCl + 2CH_3COOH \rightarrow [P]\text{-}SO_2NCl_2 + 2CH_3COONa + H_2O \quad (7)$$

As a starting material Wofatit KS-10 (produced by Chemie AG Bitterfeld Wolfen, Germany)—a commercially available sulfonate cation exchanger was used. This is a macroporous poly(S/20%DVB) resin, containing 4.2 mmol/g $–SO_3H$ groups in the dry state (sphere-like, 0.3–1.2 mm in diameter, pore volume 0.35–0.50 cm^3/g, specific surface 20–30 m^2/g).

The product—*N*,*N*-dichlorosulfonamide resin—contained 2.02 mmol/g $–SO_2NCl_2$ groups (i.e. 4.0 mmol active chlorine/g or 8.0 meq active chlorine/g) and a small amount of sulfonic groups (0.7 mmol/g $–SO_3Na$ groups). The presence of hydrophilic sulfonic groups in reactive polymers is advantageous because the sulfonic groups facilitate reactions of DCSR with water soluble reagents. The product had the form of light yellow opaque spheres. The bulk density of the air dried DCSR was 0.61 g/cm^3. The swelling in water was 10%. Details of the preparation and characterisation of the DCSR were reported elsewhere [4].

2.2. Cyanide solutions

Potassium cyanide GR—produced by E. Merck, Germany was applied for preparation of aqueous cyanide solutions used for testing. These solutions contained potassium cyanide and sodium hydroxide in different concentrations:

(a) solutions used in experiments carried out in a static system:

0.001 M KCN (i.e. 26 mg CN^-/dm^3) in 0.01, 0.1 and 1 M NaOH
0.01 M KCN (i.e. 260 mg CN^-/dm^3) in 0.03, 0.1 and 1 M NaOH
0.1 M KCN (i.e. 2600 mg CN^-/dm^3) in 0.3 and 1 M NaOH

(b) solution used in experiments carried out in a dynamic system:

100 mg CN^-/dm^3 in 0.01 M NaOH
35 mg CN^-/dm^3 in 0.0025 and 0.005 M NaOH

2.3. Analytical methods

To determine the concentration of cyanide ions in solutions two methods were employed:

- an argentometric potentiometric titration with 0.01 M $AgNO_3$ (when concentration of cyanide ions was above 100 mg CN^-/dm^3),
- a colorimetric method using barbituric acid and pyridine, spectrophotometer Spekol, Carl Zeiss Jena (when concentration of cyanide ions was in the range of 0.05–100 mg CN^-/dm^3) [8,9].

The Merckoquant cyanide test produced by E. Merck, Germany—a rapid guiding test for the detection and semi-quantitative determination of cyanide ions (1–30 mg CN^-/dm^3) was also used.

Argentometric potentiometric titration was carried out with the use of Ag/AgCl-calomel electrode system. This method makes it possible to determine CN^- and Cl^- ions simultaneously. The potentiometric titration curve has three points of inflection according to the formation of $Ag(CN)_2^-$, AgCN and AgCl. The very low potential of cyanide ions (below −500 mV) in relation to the initial potential of chloride ions (above +150 mV) is useful for monitoring effluents in a flow system.

The presence of cyanate ions in the reaction mixture was identified by the Werner test using pyridine, 1% aqueous solution of copper sulphate and chloroform. During the test a lilac-blue colour appears in the chloroform layer due to the formation of the complex $Cu(OCN)_2(C_5H_5N)_2$ [9,10]. Carbonate ion and ammonia concentration in examined post-reaction solutions were determined according to the standard methods.

The total sulphur and nitrogen contents in the resin was determined by elemental analysis (CHNS EA-1110, CE Instruments). The active chlorine content in it was determined by the iodometric method [4].

2.4. Treatment of cyanide solutions

Experiments on decomposition of cyanide ions by the *N*,*N*-dichlorosulfonamide resin were carried out batchwise and in a flow system at room temperature (18–20°C), because of unstability of that resin at higher temperature [4].

In preliminary investigations carried out in static conditions known quantities of DCSR (0.25–1.0 g) were added to a known volume of cyanide solution in an iodine flask containing enough NaOH to maintain pH >10. The reagents—active chlorine in resin and cyanide ion in solution—were used in various mole ratios. Excess of active chlorine or cyanides in relation to stoichiometry of reaction (1) was employed alternatively. The flasks were agitated using shaker. Time dependent measurements were made. Solution samples were taken after fixed time intervals and analysed. After reaction the polymeric reagent was separated from the reaction medium by filtration and was analysed (active chlorine, elemental analysis, IR).

During the experiments carried out in the dynamic system a sample of DCSR (9 g in a dry state, 16 cm^3 after swelling in water) was packed into a glass column (id 1.15 cm, height of packing ~20 cm). The cyanide aqueous solutions containing 100 mg CN^-/dm^3 in 0.01 M NaOH or 35 mg CN^-/dm^3 in 0.0025 or 0.005 M NaOH were passed through the column. The flow rate was in the range 1–5 cm^3/min (nearly 4–20 bed volumes/h). The samples of effluent (100 cm^3) were analysed.

3. Results and discussion

The results of the preliminary investigations carried out in batch method are summarised in Figs. 1–4 and

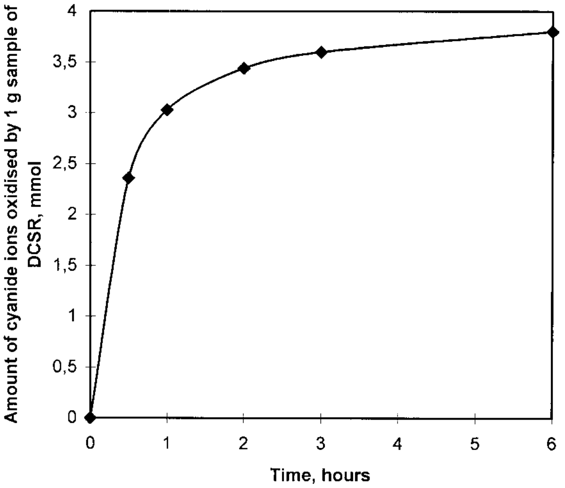

Fig. 1. Decomposition of cyanide ions (excess of cyanides) in aqueous solution during batchwise contact with a sample of *N*,*N*-dichlorosulfonamide resin, 1.0 g DCSR, 60 cm^3 0.1 M KCN in 1 M NaOH (mole ratio of active chlorine in DCSR : KCN in solution 1 : 1.5).

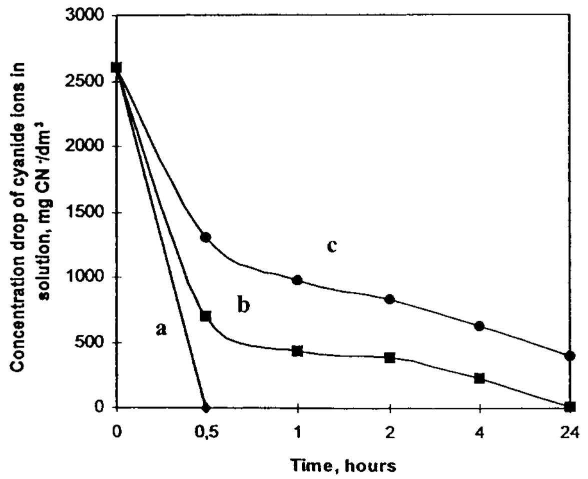

Fig. 2. Decomposition of cyanide ions (excess of active chlorine) in aqueous solution during batchwise contact with a sample of *N*,*N*-dichlorosulfonamide resin, 1 g DCSR and (a) 15 cm^3 (b) 25 cm^3 (c) 40 cm^3 0.1 M KCN in 1 M NaOH (mole ratio of active chlorine in DCSR : KCN in solution (a) 1 : 0.375, (b) 1 : 0.675, (c) 1 : 1).

Table 1. In the case of using the excess of cyanides the reaction between a sample of DCSR and 0.1 M KCN in 1 M NaOH was finished after a few hours (Fig. 1). The reaction mixture lost nearly 3.85 mmol of cyanide ion (i.e. 64% of the initial amount). All active chlorine left the resin phase and was found as chloride in the solution, whereas the resin functional groups were transformed to sulfonamide groups. These results showed that 1 mol of active chlorine reacts with 1 mol of cyanide ion (i.e. 1 mol of *N*,*N*-dichlorosulfonamide groups reacts with 2 mole of cyanide ions). This indicates that the reaction proceeds according to reaction (1). The analogous reaction carried out in a less alkaline medium (in 0.3 M NaOH) showed a similar kinetic course.

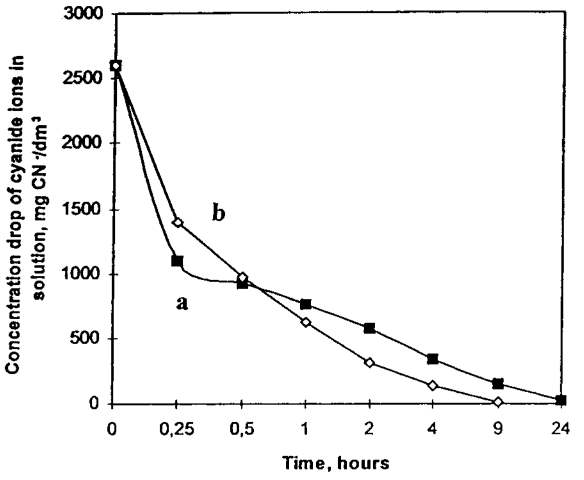

Fig. 3. Influence of the basicity of the reaction medium on decomposition of cyanide ions in aqueous solution during batchwise contact with a sample of *N*,*N*-dichlorosulfonamide resin, 1 g DCSR and 30 cm^3 0.1 M KCN in (a) 1 M NaOH, (b) 0.3 M NaOH (mole ratio of active chlorine in DCSR : KCN in solution 1 : 0.75).

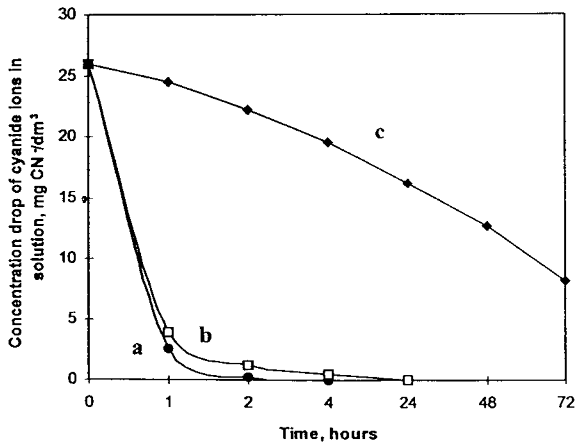

Fig. 4. Influence of the basicity of the reaction medium on decomposition of cyanide ions in dilute aqueous solution during batchwise contact with a sample of *N*,*N*-dichlorosulfonamide resin 0.25 g DCSR and 500 cm^3 0.001 M KCN in (a) 1 M NaOH, (b) 0.1 M NaOH, (c) 0.01 M NaOH (mole ratio of active chlorine in DCSR : KCN in solution 1 : 0.5).

Fig. 2 shows the course of the oxidation of different volumes of cyanide solution (0.1 M KCN in 1 M NaOH) by 1 g samples of DCSR. In the first case (15 cm^3 cyanide solution, curve a) after ca. 30 min the cyanide ions were absent in reaction medium (concentration CN^- ion fell below 0.05 mg/dm^3). In the second case (25 cm^3 cyanide solution, curve b) after 30 min the drop of these ions reached 80% in reference to the starting value, thereafter during a few hours the remaining amount of cyanide ions was destroyed. In the third case (40 cm^3 cyanide solution, curve c) after 30 min the reaction medium contained a half of the starting value of cyanide ions, whereas the remaining amount decomposed in a few days.

The next step of the experiments was the oxidation of 0.1 M KCN solution by a sample of DCSR in more strongly alkaline medium (1 M NaOH) and in less alkaline medium (0.1 M NaOH) (Fig. 3). After 30 min in both cases nearly 2/3 of the starting amount of cyanide ions was oxidised, the residual 1/3 of its amount was oxidised far slower. From this figure it seems that the first part of the reaction goes rather faster in 1 M NaOH solution, whereas the second part of the reaction goes rather faster in 0.1 M NaOH solution.

A detailed description of the influence of basicity of the reaction medium on the kinetic course of the oxi-

Table 1
Concentration drop of cyanide ions in solution during batchwise contact with a sample of *N*,*N*-dichlorosulfonamide resin

Time (h)	(a) 0.5 g DCSR, 100 cm^3 0.01 M KCN (mole ratio of active chlorine in DCSR : KCN in solution 2 : 1)			(b) 0.25 g DCSR, 100 cm^3 0.001 M KCN (mole ratio of active chlorine in DCSR : KCN in solution 1 : 0.1)		
	0.03 M NaOH	0.1 M NaOH	1 M NaOH	0.01 M NaOH	0.1 M NaOH	1 M NaOH
0	260.0	260.0	260.0	26.0	26.0	26.0
0.25	–	–	13.0	19.3	< 0.05	< 0.05
0.50	–	–	6.7	14.7		
1	152.5	95.2	1.2	8.8		
2	123.4	41.0	< 0.05	0.05		
4	66.7	10.2				
8	30.6	2.6				
24	16.4	1.1				
	Active chlorine in sample of DCSR (mmol)					
Before reaction	2.0	2.0	2.0	1.0	1.0	1.0
After reaction	0.96	0.94	1.02	0.89	0.69	0.62

dation of cyanide ions by DCSR is given in Table 1. It is evident that the reaction goes rapidly in strong alkaline medium (in 1 M NaOH solution) and slowly in weak alkaline medium (in 0.03 M NaOH) (part (a) of this table). Examining the remaining polymeric reagent after kinetic investigations it was found that in all cases the active chlorine content in resin fell and the drop was approximately adequate to the cyanide ions content in the starting solution.

The data of the reactions between a sample of DCSR and the solution sample containing a small amount of cyanide ions are given in part (b) of this table. In this case one can observe a similar relationship—the reaction goes fast in more alkaline solutions (in 1 M NaOH and 0.1 M NaOH) and slowly in less alkaline medium (in 0.01 M NaOH). Examining the remaining polymeric reagent after kinetic investigations it was found that the active chlorine content in resin fell—but the drop was larger than it results from stoichiometry of reaction (1). It appears that DCSR loses a part of active chlorine in strong alkaline media as a result of *N*,*N*-dichlorosulfonamide groups decomposition to *N*-chlorosulfonamide groups:

$$[P]\text{-}SO_2NCl_2 + 2NaOH \rightarrow [P]\text{-}SO_2NClNa + Na^+ + OCl^- + H_2O \qquad (8)$$

Active chlorine which quits the resin phase is found as chloride in the solution:

$$OCl^- \rightarrow Cl^- + [O] \qquad (9)$$

$$2[O] \rightarrow O_2 \qquad (10)$$

In the last series of experiments which were carried out in a static system dilute cyanide solutions (0.001 M KCN) in sodium hydroxide in different concentrations and excess of active chlorine was used (Fig. 4). The reaction course in 1 M NaOH and in 0.1 M NaOH (curve a, b) differs from the reaction course in 0.01 M NaOH solution (curve c). Fig. 4 shows that only in a weakly alkaline medium ($pH < 12$) both atoms of active chlorine are relatively strongly bound by the sulfonamide groups and the reaction goes on gradually.

The preliminary investigations carried out in a flow system consist in passing through the column a solution containing 100 mg CN^-/dm^3 0.01 M NaOH. An exothermic effect of reaction and the liberation of a gaseous product (colourless, odour-free gas, i.e. oxygen, formed according to reactions (9) and (10)) in the column was stated. Although the concentration of cyanide ions in effluent was greatly reduced (below 0.05 mg CN^-/dm^3)—a bulk concentration of chloride ion was found in it. The concentration of chloride ion in effluent exceeded greatly the results of reaction (1) stoichiometry. The quick inactivation of the resin as a result of the loss of active chlorine eluted from the resin bed by sodium hydroxide as well as the resistance to flow through, caused by the liberation of gaseous products do the described above procedure inefficient for cyanide ions decomposition in a flow system.

Using the results of experiments carried out in static condition attempts were made to pass through the resin bed less alkaline cyanide solution. This tested solution contained 35 mg CN^-/dm^3 in 0.005 M NaOH. The data for cyanide ion decomposition are shown in Fig. 5a. Effluent was controlled continuously using Ag/AgCl–calomel electrode system. When the redox potential of effluent dropped below $+50$ mV, the cyanide ions concentration was determined by colorimetric

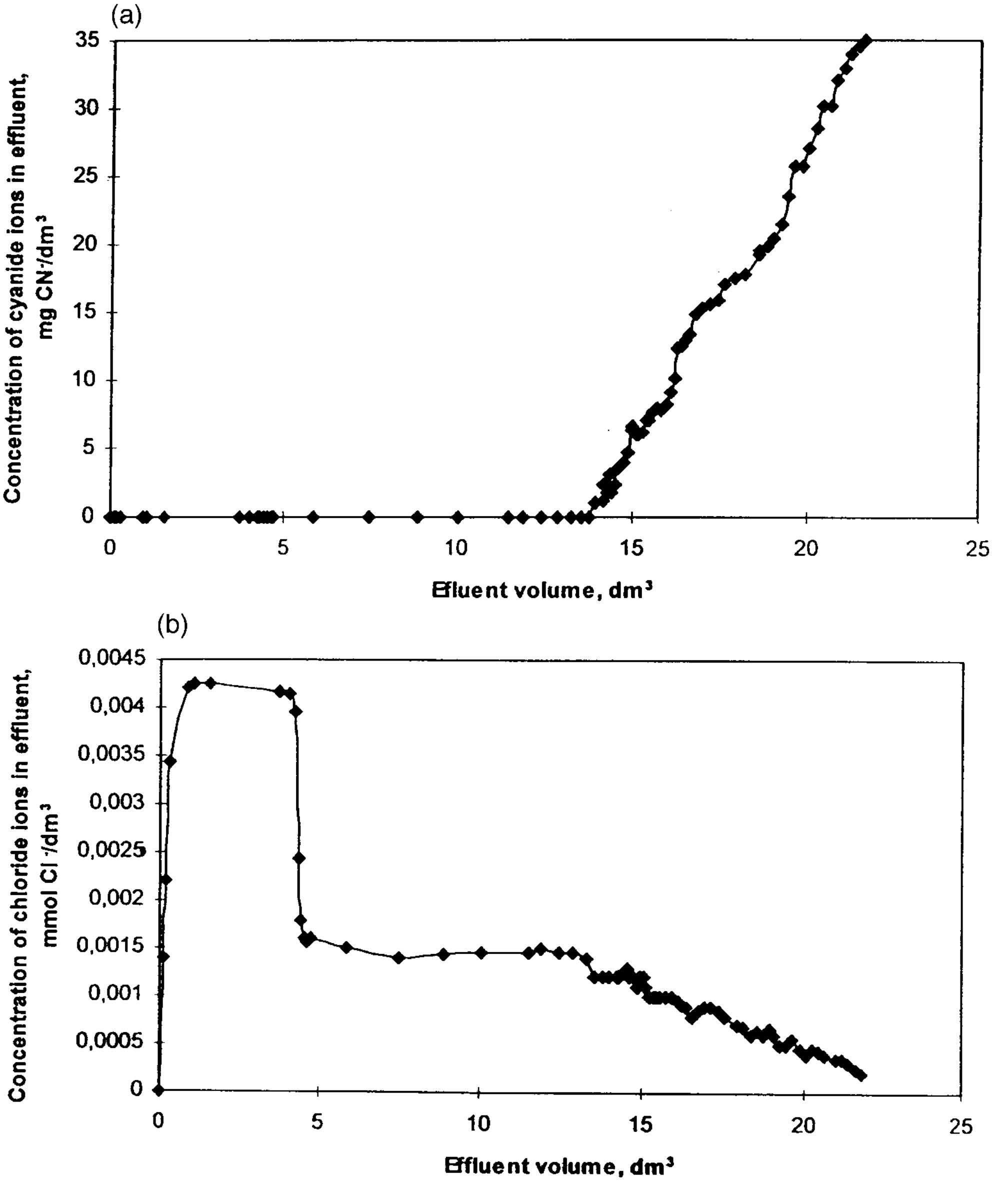

Fig. 5. Decomposition of cyanide ion in a flow system. Bed volume of DCSR ~16 cm^3, height of packing ~20 cm. Influent concentration: 35 mg CN$^-$/dm^3, 0.20 g NaOH/dm^3. Flow rate: 7.5 bed volumes/h. (a) cyanide break-through curve, (b) concentration of chloride ions in effluent.

analysis (this value follows from measurements of the redox potential of dilute cyanide aqueous solutions and from the course of the potentiometric 0.01 M $AgNO_3$ titration curve CN$^-$ and Cl$^-$ ions). From Fig. 5a it is evident that the concentration of cyanide ions in effluent was greatly reduced. At the initial stage of these studies the effect of flow rate on the oxidation was tested. The loss of cyanide ions in effluent depends on the residence time of the feed in the reactor bed. When the flow rate was about 7.5 bed volumes/h the concentration of CN$^-$ ions in effluent was below 0.05 mg/dm^3 (ca. 700-times less than the initial concentration). Cyanide column break-through started after 14 dm^3 (nearly 950 bed volumes). The amount of active chlorine in resin bed (36 mmol) is higher than the amount of cyanide ions decomposed during the column experiments (18 dm^3 × 1.35 × 10^{-3} M CN$^-$/dm^3 = 24 mmol). The reason of this difference is that in the beginning of the column-test the effluent contains much more chloride ions than it results from stoichiometry of reaction (1) (Fig. 5b). The course of the break-through curve indicates that cyanide ions were more effectively decomposed by the action of the *N*,*N*-dichlorosulfonamide resin at the time when the reaction was carried out in less alkaline medium than previously. Only a small amount of liberated gaseous products was observed in that case.

The third cyanide test solution decomposed in the flow system with the use of DCSR contained 35 mg CN^-/dm^3 in 0.0025 M NaOH. Cyanide column break-through started after 22.4 dm^3 (near 1500 bed volumes). The amount of active chlorine in the resin bed (36 mmol) was nearly adequate to the amount of cyanide ions decomposed in the column test ($25.2\ dm^3 \times 1.35 \times 10^{-3}$ M $CN^-/dm^3 = \sim 34$ mmol). The liberation of gaseous products in the column during the dilute cyanide solution flow could not be observed. The chloride ion concentration in effluent had approximately a constant level of nearly 50 mg Cl^-/dm^3.

As it was stated earlier the reactive polymer containing active chlorine in functional groups reacts with potassium cyanide forming potassium cyanate [1]. The presence of cyanate ion in effluents was determined qualitatively. As mentioned above the alkali metal cyanates (1000-times less toxic than cyanides) are unstable in aqueous solutions—they hydrolyse to carbonates and ammonia:

$$OCN^- + 2H_2O \rightarrow HCO_3^- + NH_3 \qquad (11)$$

The presence of cyanate hydrolysis products in post-reaction media was confirmed.

After the third column test DCSR was water-washed, treated with 0.1 M HCl, again water-washed, air-dried and analysed. It was proved that the copolymer after cyanide solution treatment lost nearly all active chlorine. It contained 3.7 mmol/g nitrogen and 0.8 mmol/g sulfonic groups (freshly prepared sulfonamide copolymer contains 3.9 mmol/g nitrogen). To identify the chemical structure of the used copolymer its IR spectrum and the spectrum of a freshly prepared sulfonamide copolymer were compared. It was observed that the IR spectrum of the used copolymer showed characteristic SN and NH_2 vibration bands [4].

The exhausted copolymer can be regenerated and reused for continuous processes. It was found that after rechlorination the product contained 7.6 meq/g of active chlorine (nearly 95% amount of active chlorine introduced in the first cycle).

4. Conclusions

1. The results obtained in this work indicate that a copolymer having *N*,*N*-dichlorosulfonamide groups is useful in treating cyanide aqueous solutions in order to destroy CN^- ions, however its action differs from that of *N*-monochlorosulfonamide resin. Both chlorine atoms contained in the *N*,*N*-dichlorosulfonamide group show oxidative properties in relation to cyanide ions, but their reactivity is different in examined conditions.
2. The second active chlorine atom attached to *N*-chlorosulfonamide group forming *N*,*N*-dichlorosulfonamide group—easily quits the resin phase in strong alkaline media ($pH > 12$) and quickly oxidises the CN^- ions contained in solution. In many experiments more active chlorine quits the resin phase than it results from stoichiometry of the reaction (1). Therefore cyanide solutions having high alkalinity can not be treated in flow system (the inefficient utilisation of active chlorine, perturbation in resin bed as an effect of a gaseous product forming (oxygen) in reaction medium).
3. To utilise with good efficiency both of the two active chlorine atoms attached to the macromolecular support for destruction of cyanide ions carried out in flow system it is necessary to maintain the level of basicity of the influent as low as possible ($pH < 11$).
4. Owing to very high concentration of active chlorine in resin bed (2.15 mole of active chlorine/dm^3) it is possible to obtain an effluent free of cyanides.
5. Cyanide ions being oxidised by DCSR transform to cyanates which are far less toxic that cyanides. Cyanate are unstable in aqueous solutions—they hydrolyse to ammonia and carbonates. In spite of very high concentration of active chlorine in the resin bed the cyanate ions did not transform to nitrogen and carbon dioxide by further flowing through the column.
6. When the more reactive of the two active chlorine atoms quits the resin phase—a *N*-mono-chlorosulfonamide group results. The properties of the reactive polymer having *N*-mono-chlorosulfonamide groups in relation to cyanide ions have been investigated in an earlier report [1].
7. The reactive copolymer during the cyanide solution treatment undergoes dechlorination, i.e. the sulfonamide groups are being restored. It can be regenerated by the use of hypochlorite solutions (7).

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