



Treatment of a synthetic solution of galvanization effluent via the conversion of sodium cyanide into an insoluble safe complex

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ABSTRACT

Wastewater discharged from metal-finishing processes usually contains cyanide, a hazardous substance that is used extensively in the surface finishing industry. In the present study, a synthetic solution resembling the contaminated wastewater was chemically treated using ferrous sulfate. This simple one-step process was applied successfully to remove cyanide from metal finishing wastewater. The experiments were carried out on a synthetic solution containing ions of cyanide and zinc. The effects of mixing velocity, ratio of ferrous ions to cyanide ions, ferrous ions concentration, initial cyanide concentration, pH of solution, temperature, mixing time and zinc ions concentration were studied. The results showed that the removal efficiency of cyanide increased as the mixing velocity increased, ferrous ion molar ratio to cyanide ions increased, temperature increased and time of mixing increased at an optimum of pH 8. The reduction of cyanide concentration reached the allowable limit for wastewater discharge according to the Egyptian Environmental Law decree 44/2000, which is 0.2 mg/l. The formed complexes were analyzed and the stability of each complex was studied under different pH value solutions after 7 days of contact. A typical example of electroplating wastewater from a local company in the field of metal finishing, which contains 18 mg/l CN^- and 12 mg/l Zn^{2+} , was treated according to the determined optimum conditions for the treatment process and the concentration of CN^- was reduced to 0.095 mg/l after 15 min of agitation.

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1. Introduction

Cyanide, a poisonous substance dangerous to humans, animals, plants and aquatic organisms, can be found in the effluent of several industries such as paint and ink formulation, petroleum refining, explosives, case hardening, automobile industry, chemicals industry, pesticides industries, synthetic fiber production, electroplating, thermoelectric power stations, mining, electronics, and coke [1–18]. Its effects on the human and the environment have been well reviewed by Mudder and Botz [14]. Due to its toxic nature, cyanide must be recovered, removed or destroyed. There are many reported processes for treating cyanide-containing effluents [1–30]. These processes can be categorized as biodegradation [1,24,26]; adsorption on activated carbon [6,18]; oxidation via chemical, electrochemical or photochemical processes [7,9,10,12,15,19,23]; chemical precipitation [11]; hollow fiber gas membranes [13]; ultrasonic waves [15]; ion exchange [17]; extraction [20]; photocatalytic using Ti catalyst [22,25]; and volatilization then absorption by NaOH [21]. The suitability of any

of the above-mentioned processes to a specific cyanide-containing effluent depends on the effluent flow rate, cyanide concentration, associate chemical species, permissible level of cyanide in the effluent after treatment, technical level of the entity's employees and the economy and finances of the process.

In Egypt, electroplating workshops are major sources for cyanide-contaminated wastewater. They are geographically distributed all over the country. Most of them are small size entities operated by a few low technical level labors. Due to these uncomfortable circumstances, it is not possible to individually treat the effluent of every workshop using an advanced technique or to collect the effluent of these small entities to be treated in a centralized treatment unit. Currently, most of these workshops do not treat their waste by any means. They discharge the effluents as is to the nearest sewage line creating a severe environmental problem. A real case study of a medium-size Egyptian electroplating workshop producing effluent of 31 m³/day was presented by Abou-Ellela et al. [8]. They treated the cyanide-containing effluent using alkaline chlorination, which does not destroy the pollutants completely [27]. In caustic chlorine treatment, cyanide is converted to cyanate which is also toxic. Complete destruction of cyanate is difficult. The chlorination process also produces secondary by-products such as trihalo-methanes, which are highly toxic and carcinogenic [28]. The treated water contains huge amount of dissolved chlorine. Considering the nature of the cyanide effluents of the Egyptian

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electroplating industry, there is a need for another treatment method, which can completely remove cyanides. Besides, the process to be adopted must have two other criteria: (1) economic for small-scale operation and (2) easy to handle by unskillful labor.

In this work, we recommend ferrous sulfate addition to the cyanide-containing wastewater as a simple and efficient treatment process. The use of an iron salt is appropriate for all cyanide concentrations, slurry application and solution application. Ferrous sulfate also has the advantage of low cost and wide availability, as it is generated from the steel pickling in the metal surface finishing industry. The effects of mixing velocity, time of stirring, pH, concentration of ferrous ions and its molar ratio to cyanide ions, and the temperature of reaction on the removal process were investigated. The elemental analysis of the precipitated complex and its stability in solutions with varying pH value has been also studied.

2. Experimental work

2.1. Materials

The chemicals used during this work were pure grade. Sodium cyanide, ferrous sulfate and zinc oxide were supplied by United Co., while sodium hydroxide and sulfuric acid were supplied by ADWIC Co. Distilled water was used to prepare the solution and to wash the glassware in each experimental run.

2.2. Instrumentation

All samples and chemicals used in the preparation of solutions were weighed using an analytical balance of “Sartorius” type with a sensitivity of 10^{-4} g.

Mixing was performed using J. P. Selecta magnetic stirrer whose speed can be varied from 100 to 1600 rpm. The pH of solutions was measured by using Inolab pH meter. The cyanide is the key parameter selected for determining the treatment efficiency. It was measured using 975-MP Spectrophotometer, which is obtained from ORBECO Analytical Systems, Inc. Kits 975 MP/32 were used during measurements. Elemental analyses of Zn and Fe were performed using GBC 902 Atomic Absorption, while the analyses of C and N were done using PerkinElmer CHNS/O elemental analysis.

2.3. Preparation of cyanide solutions

In this study, the cyanide solution was prepared by dissolving 0.2 g of sodium cyanide 96% purity and 0.16 g of zinc oxide 90% purity in a small amount of distilled water. Then the solution was transferred to a measuring flask and distilled water was added to make up a total volume of 1000 ml.

2.4. Preparation of ferrous sulfate solution

The ferrous sulfate solution was prepared by dissolving 2 g of ferrous sulfate 90% purity in a small amount of distilled water. Then the solution was transferred to a measuring flask and distilled water was added to make up a total volume of 1000 ml.

2.5. Experimental procedure

A known volume of ferrous sulfate is added to a known volume of cyanide solution in a glass flask. The solution was mixed at a certain RPM for a certain time. The solution was then filtrated using a quantitative filter paper. The filtrate was then analyzed for cyanide concentration, while the precipitated complex was elementally analyzed using atomic absorption and CHNS/O analyzer. This procedure was used to obtain the optimum mixing intensity,

$\text{Fe}^{2+}/\text{CN}^-$ molar ratio, pH, Fe^{2+} concentration, time, temperature and $\text{Zn}^{2+}/\text{CN}^-$ molar ratio.

2.6. Cyanide determination

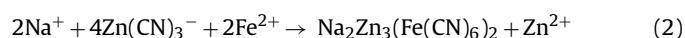
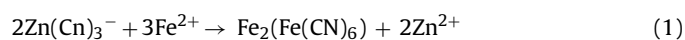
The analysis of cyanide is not an easy task as it is subjected to various types of interferences. Different analysis techniques have been reported such as colorimetric techniques using a spectrophotometer after cyanides distillation [6,11,19], potentiometric techniques using silver electrode [18], titration method using silver nitrate and 4-dimethylaminobenzylidinerhodanine indicator [19,21], ion pair/ion interaction chromatography (IIC) [29,30], the EPA Standard Method 335.4 [31] and the EPA Standard Method OIA-1677 [32].

Although, the above-mentioned techniques give reliable results, we preferred to use the simple colorimetric method using ORBECO spectrophotometer with a pretested chemical kit as it is a straightforward method. The aim is to reach a simple procedure suitable for being adopted by the local electroplating workshops labor. Any analysis method that involves the liberation of HCN was discarded for safety reasons.

3. Results and discussions

3.1. Analysis of the formed complex

Elemental analyses for the formed complex have been done using the instruments described above. Table 1 shows the weight percentage of every element. The results of these analysis showed that the atomic ratios of Na, Zn, Fe, C and N are 2, 3, 5.4, 14.5 and 14.5, respectively. Although it is not easy to determine the structure of the formed precipitate based on the elemental analysis only, we can only suggest that the precipitate that forms in the reaction between Fe^{2+} and CN^- in case of presence of Zn^{2+} and Na^+ ions is comprising mainly insoluble sodium–zinc hexacyanoferrate $\text{Na}_2\text{Zn}_3[\text{Fe}(\text{CN})_6]_2$ and iron ferrocyanide $\text{Fe}_2[\text{Fe}(\text{CN})_6]$ according to the reactions below:



3.2. Effect of mixing velocity

In this set of experiments, the mixing velocity was varied between 100 and 1600 rpm. The concentration of the cyanide in the solution was measured as a function of mixing intensity, as shown in Fig. 1, which shows the effect of mixing velocity on cyanide removal at different mixing times. The increase in the cyanide removal is due to the increase in the rate of collisions between reactant species, which finally leads to the increase of the rate of reaction. An appreciable increase in the rate of reaction occurs as mixing velocity and time increase, which is due to the increase in contact time between species of reactants, which finally leads to the increase the rate of reaction. Mixing velocity of 1600 rpm with 30 min of mixing gave the highest efficiency for cyanide removal process.

Table 1
The elemental analysis (%) for the formed complex.

Na	4.17
Fe	30.44
Zn	19.72
C	17.4
N	20.29

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