

A comparison of pilot scale electron beam and bench scale gamma irradiations of cyanide aqueous in solution (Postprint)

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Abstract

Bench-scale gamma irradiation was conducted to investigate the influencing factors, such as initial cyanide concentration, absorbed dose, saturated gases, and pH value as well as water compositions on the γ radiolysis of cyanide in simulated aqueous solution. The decomposition rate of cyanide was observed to follow pseudo-first-order kinetics over the applied concentration range of 77 mg/L to 247 mg/L. Cyanide was decomposed more rapidly at lower initial concentrations than at higher initial concentrations. However, radical scavengers in natural waters, such as carbonate and bicarbonate, have negative effects on cyanide removal. This indicated that hydroxyl radicals may play a predominant role in the γ radiolysis of aqueous cyanide. Finally, ammonia and cyanate were identified as the main nitrogen-containing byproducts of γ radiolysis of cyanide. To remove toxic hydrogen cyanide (HCN) from carbon fiber industry waste gases, a pilot-scale experiment with a self-sheltered electron beam accelerator was demonstrated after two sprays of chemical absorption. The operating conditions for absorption and irradiation were optimized. It was shown that after the first spray tower, HCN concentration decreased from $(240 \pm 50) \text{ mg/m}^3$ to $(35 \pm 15) \text{ mg/m}^3$. While after the second spray tower, effluent HCN was reduced almost below concentration was controlled at $(15 \pm 2) \text{ mg/L}$ with a water regulating tank. This treatment allowed CN^- to reach the regional limit (of 0.5 mg/L) for safe industrial wastewater discharge with an irradiation dose of 12 kGy. The obtained results showed that the combined process was effective for removing HCN from the waste gas.

Full Text

Preamble

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A Comparison of Pilot-Scale Electron Beam and Bench-Scale Gamma Irradiations of Cyanide in Aqueous Solution

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Abstract

Bench-scale gamma irradiation was conducted to investigate the influencing factors on the γ -radiolysis of cyanide in simulated aqueous solution, including initial cyanide concentration, absorbed dose, saturated gases, pH value, and water composition. The decomposition rate of cyanide followed pseudo-first-order kinetics over the applied concentration range of 77 mg/L to 247 mg/L. Cyanide decomposed more rapidly at lower initial concentrations than at higher ones. However, radical scavengers present in natural waters, such as carbonate and bicarbonate, negatively affected cyanide removal, indicating that hydroxyl radicals may play a predominant role in the γ -radiolysis of aqueous cyanide. Ammonia and cyanate were identified as the main nitrogen-containing byproducts of cyanide radiolysis. To remove toxic hydrogen cyanide (HCN) from carbon fiber industry waste gases, a pilot-scale experiment with a self-shielded electron beam accelerator was demonstrated after two stages of chemical absorption. The operating conditions for absorption and irradiation were optimized. After the first spray tower, HCN concentration decreased from $(240 \pm 50) \text{ mg/m}^3$ to $(35 \pm 15) \text{ mg/m}^3$. After the second spray tower, effluent HCN was reduced almost below the method detection limit. The residual cyanide concentration was controlled at $(15 \pm 2) \text{ mg/L}$ using a water regulating tank. This treatment allowed CN^- to reach the regional limit of 0.5 mg/L for safe industrial wastewater discharge at an irradiation dose of 12 kGy. The obtained results showed that the combined process was effective for removing HCN from waste gas.

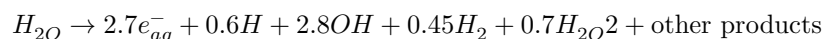
Keywords: Gamma irradiation, Electron beam, Wastewater treatment, Cyanide, Ozone, Advanced oxidation processes

Introduction

Cyanide and other cyano-compounds that liberate free cyanide ions (CN^-) are highly toxic to almost all forms of fauna. They bind with cytochrome oxidase to inhibit its activity and induce cellular anoxia. Cyanide is widely used in metal cleaning, electroplating, steel tempering, ore leaching, metal processing, and other industries, making remediation of cyanide-contaminated gas and water necessary. Typical treatment methods include alkaline chlorination, hydrogen peroxide oxidation, ozonation, and biodegradation, but all these processes have

several disadvantages. Biological oxidation may require long adaptation periods and cannot treat high concentrations effectively. Chlorine can react with organics to form chlorinated compounds and may result in huge amounts of total dissolved solids (TDS) in the treated water. Hydrogen peroxide successfully oxidizes cyanides only in the presence of copper as a catalyst, which must subsequently be separated and recycled from the purified wastewater. Ozone treatment imposes high reagent costs. The SO₂/air process is relatively inexpensive, but some reagent savings are offset by license/royalty payments. Consequently, developing a more cost-effective and environmentally friendly treatment process is desired.

The positive effects of ionizing radiation processing for environmental protection are well known. Current applications include radiation treatment of wastewater, sewage sludge, flue gases, and solid wastes. The principles of radiation treatment of wastewater can be explained by reaction (1). In reaction (1), hydroxyl radicals ($\cdot\text{OH}$, $E^0=2.8\text{V}$) are the strongest oxidizing species, while hydrated electrons (e_{aq}^- , $E^0=-2.8\text{V}$) and hydrogen atoms ($\cdot\text{H}$, $E^0=-2.1\text{V}$) are the main reducing species in water radiolysis. In most radiolysis solutions, these reactive primary species (e_{aq}^- , $\cdot\text{H}$, and $\cdot\text{OH}$) initiate the decomposition of water pollutants.



The objective of bench-scale ⁶⁰Co γ -source experiments was to investigate gamma irradiation-induced degradation kinetics of cyanide at different initial concentrations. Various influencing factors important to cyanide decomposition were studied, including absorbed dose, initial cyanide concentration, saturated gases, initial solution pH, and water composition. Moreover, the principal reaction byproducts of γ -radiolysis of cyanide, such as ammonia and cyanate, were quantitatively examined. Pilot-scale electron beam irradiation was demonstrated to explore the feasibility of combined technology for eliminating toxic cyanide-containing gas from Zhong Fu Shen Ying Carbon Fiber Ltd., Jiangsu Province, China. The results suggest that treating cyanide waste gas by combined chemical absorption and radiation degradation is feasible and demonstrate good potential for ionizing radiation processing applications in environmental protection in China.

2.1 Irradiation Experiments

Bench-scale experiments were performed using a gamma irradiator located at the Institute of Nuclear and New Energy Technology (INET), Tsinghua University. The ⁶⁰Co γ -source holder consists of 16 source rods with a total activity of about 4.8×10^{14} Bq. In each irradiation experiment, 25 mL of cyanide solution was transferred to 50 mL Pyrex glass bottles and placed in the ⁶⁰Co irradiation field at specific distances and times to achieve absorbed doses ranging from 5 to 35 kGy. The absorbed dose rate used in this study was fixed at 279 Gy/min, determined by means of a Fricke dosimeter using $G(\text{Fe}^{3+}) = 15.6$.

Pilot plant irradiation was operated with a self-shielded electron beam accelerator (EA, energy 0.5–1.0 MeV, beam current 10–15 mA), manufactured by Jiangsu Dasheng Electron Accelerator Device Co., Ltd. Aqueous streams of wastewater containing cyanide were exposed to a scanned beam in a water film from two nozzles. Each nozzle was designed to be 10 cm wide and 0.2 cm thick according to the penetration depth of radiation in water. The average speed of the water film through the beam was approximately 1.37 m/s, yielding a retention time of approximately 0.05 s and a flow rate of about 1.82 m³/h. The irradiation dose was calculated using an empirical formula described by Luo and Xu. All experiments were carried out at ambient temperature.

2.2 Materials

KCN and other reagents were obtained from Beijing Chemical Co. Inc., China. All chemicals were of analytical grade or the highest purity available from the supplier. Sample solutions of cyanide were prepared in doubly distilled water or in tap water from INET as a control. Operators were provided with personal masks and portable HCN gas monitor/alarm units. To investigate the effects of different saturated gases in radiolysis, nitrogen and oxygen gas were continuously bubbled into the aqueous solution during irradiation at a flow rate of 0.5 L/min, respectively. The purity of nitrogen and oxygen gases was more than 99.99%.

2.3 Analytical Methods

The concentration of CN⁻ was determined by titration with silver nitrate standard solution and by the nicotinic acid and 1-phenyl-3-methyl-5-pyrazolone photometric method. Changes in solution pH during the irradiation process were recorded using a WTW pH meter. Ammonia (measured as NH₄⁺) appeared to be one of the degradation products, and its yield was determined using Nessler's reagent by measuring absorption at $\lambda=420$ nm. Cyanate, another byproduct, was converted to ammonia by acid hydrolysis (Kjeldahl digestion), and total ammonia was determined using Nessler's reagent. The difference in ammonia concentration before and after acid hydrolysis represents the cyanate content. The pH of wastewater was adjusted using sulfuric acid and sodium hydroxide.

2.4 Pilot Plant Technical Process

The technical process is shown in [Figure 1: see original paper]. After preliminary gas purification, the cyanide gas passed through two spray towers in series to ensure the treated gas complied with national discharge standards (1.9 mg/m³). HCN was entrapped in an absorption solution containing sodium hypochlorite. The residual cyanide in aqueous solution was then introduced into a process vessel and irradiated by a combined process of electron beams and ozonation.

3.1.1 Effects of Initial Concentrations

A series of batch experiments was conducted to evaluate the effect of initial concentrations on cyanide decomposition by gamma irradiation. Initial CN^- concentrations were 85.9 mg/L, 131 mg/L, and 247 mg/L, respectively, with an initial solution pH of 9.50 before irradiation. [Figure 2: see original paper] shows cyanide removal at different initial concentrations. The rate of CN^- degradation increased dramatically with increasing absorbed dose. At an absorbed dose of 20 kGy, the removal rates were 83.8%, 66.8%, and 38.4% at initial concentrations of 85.9 mg/L, 131 mg/L, and 247 mg/L, respectively. This observation indicated that cyanide was removed more rapidly at lower initial concentrations than at higher ones, similar to results reported for aromatic hydrocarbon and dimethyl phthalate degradation.

As shown in previous studies using electron beam or gamma irradiation, pollutant concentrations decrease exponentially with absorbed dose, which can be expressed as a first-order model:

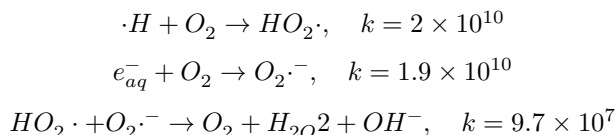
$$C = C_0 e^{-kD}$$

where C_0 and C are the pollutant concentrations before and after irradiation, respectively, k is the dose constant in units of reciprocal dose, and D is the absorbed dose necessary to achieve the specified concentration change. For convenience, Eq.(2) was modified into Eq.(3) to compare pollutant decomposition rates under various conditions.

[Figure 3: see original paper] shows the dependence of $\ln(C/C_0)$ on increasing absorbed dose. The rate constants were calculated as $1.33 \times 10^{-4} \text{ Gy}^{-1}$, $5.66 \times 10^{-5} \text{ Gy}^{-1}$, and $2.54 \times 10^{-5} \text{ Gy}^{-1}$ at concentrations of 85.9 mg/L, 131 mg/L, and 247 mg/L, respectively. The dose constant is dependent on the initial CN^- concentration and decreases significantly as concentration increases, consistent with radiolysis studies on polychlorinated biphenyls in nonpolar solution.

3.1.2 Effects of Saturated Gases

Irradiation experiments were performed under static (situation A), N_2 (situation B), and O_2 (situation C) continuous bubbling conditions. The initial cyanide concentration was 85.89 mg/L and the initial solution pH was adjusted to 10.08. In the presence of air or water saturated with pure oxygen, e^- and $\cdot\text{H}$ can react with oxygen as shown in Reactions (4)–(6). Both e^- and $\cdot\text{H}$, at low pollutant concentrations in aerated water, are converted into peroxy radicals. Depending on O_2 and cyanide concentrations in the aqueous solution, competition for e^- and $\cdot\text{H}$ between O_2 and CN^- takes place. Hence, the attacking species under these conditions are $\cdot\text{OH}$ as well as $\text{O}_2^-/\text{HO}_2\cdot$, leading to corresponding cyanide adducts.



At a dose of 5 kGy, CN^- removal efficiency was 40.96%, 47.97%, and 76.39% under situations A, B, and C, respectively. A possible explanation is that $\cdot OH$ radicals may be the main attacking species, while direct $e q^-$ and $\cdot H$ attack on cyanide plays a minor role. In situation B, excessive N_2 saturation caused O_2 concentration in the solution to drop to almost zero. However, CN^- removal efficiency was better in situation B than in situation A, possibly due to decreasing solution pH and N_2 stripping. As shown in [Figure 5: see original paper], solution pH decreased slowly from 10.08 to 9.30 with increasing absorbed dose. This situation is more conducive to HCN formation, which is then stripped off by N_2 bubbling. Similar results are reported for cyanide removal from gold leach waste solution by air stripping. It is also noteworthy that gamma irradiation can only reduce CN^- concentration to a certain extent. When radiation dose increased from 10 kGy to 20 kGy, CN^- concentration almost did not change while bubbling O_2 , but decreased slowly under the other two conditions, with removal efficiency being almost the same among all three conditions at 20 kGy.

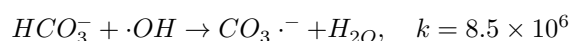
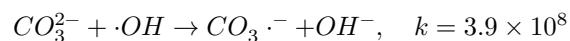
3.1.3 Effects of the Initial Solution pH

[Figure 6: see original paper] shows the dependency of initial solution pH on CN^- decomposition. The initial CN^- concentration was 85.89 mg/L, and pH before irradiation was adjusted to 9.97 and 11.7, respectively. The lower removal efficiency of CN^- at higher pH compared to lower pH may be caused by changes in active particles. This observation is similar to a previous study on 2,4,6-trinitrotoluene decomposition by gamma irradiation. Considering the earlier discussion that $\cdot OH$ radicals may be the main attacking species, a possible reason for pH effects on degradation efficiency is the acid-base properties of $\cdot OH$ radicals. Initial yields (G-values) of different reaction species produced by water radiolysis vary with solution pH. For example, the G-value of $e q^-$ decreases, $\cdot H$ increases rapidly, and $\cdot OH$ increases slightly as pH decreases below 5. Additionally, the G-values of $\cdot OH$ and $\cdot H$ decrease significantly while $O \cdot^-$ and $e q^-$ increase rapidly as pH increases above 10.

3.1.4 Effect of Water Composition

The effect of water composition on cyanide decomposition was investigated using distilled water and tap water with an initial CN^- concentration of 76.24 mg/L. [Figure 7: see original paper] shows that cyanide decomposition reached nearly 100% in distilled water at a dose of 20 kGy, while removal decreased to 69.5% in tap water at the same dose. This indicates that cyanide degradation is affected by the presence of some ions in tap water. These ions, such as carbonate and

bicarbonate, may interact with reactive species formed during irradiation as shown in reactions below:

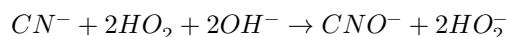
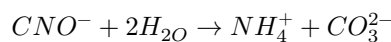


Earlier studies demonstrated that decomposition of trichloroethylene (TCE) and perchloroethylene (PCE) was significantly inhibited by these ions in natural or drinking water. According to Yoon et al., without carbonate ions, TCE and PCE decomposition increased abruptly at 100 Gy and reached 100% at 300 Gy. However, only about 20% of PCE was removed in the presence of 5×10^{-3} mol/L carbonate ions, while just 5% of TCE was removed under the same condition.

3.1.5 Analysis of Degradation Products

To study the intermediate products of γ -radiolysis of cyanide solution, experiments were carried out in a static system at pH 9.97 and an initial CN^- concentration of 85.89 mg/L. Ammonia (NH_4^+) and cyanate (CNO^-) were detected as the main byproducts rather than nitrogen gas. [Figure 8: see original paper] shows the concentrations of residual CN^- , CNO^- , and NH_4^+ as a function of absorbed dose. At the beginning of irradiation, CNO^- concentration increased sharply and reached its highest value at 5 kGy. However, with increasing absorbed dose from 5 kGy to 20 kGy, CNO^- concentration decreased slowly, possibly due to hydrolysis reactions in alkaline solution. In contrast, NH_4^+ concentration increased slowly during the entire irradiation process, reaching 32.24 mg/L at 20 kGy. Even at 20 kGy, the transformation rate from CN^- to NH_4^+ was only 57.3%.

These observations indicate that CN^- might first react with $\cdot OH$ to form $(CN)_2$, which then converts to CN^- and CNO^- in alkaline solution, with the latter hydrolyzing to NH_4^+ . Another degradation pathway might be direct oxidation by H_2O_2 and $\cdot HO_2$, as shown in Reactions (12) and (13), similar to previous studies.



However, if carbonate or bicarbonate anions were verified as the main carbon-containing byproduct of γ -radiolysis of cyanide, it can be deduced from the

discussion in Section 3.1.4 that increased carbonate or bicarbonate concentration with increasing initial cyanide concentration and absorbed dose would, in turn, inhibit cyanide radiolysis due to scavenging of hydroxyl radicals.

shows the concentration of HCN and NH_3 at different sampling points. Vaidya et al. validated that alkali chlorination with calcium hypochlorite is an appropriate treatment process for cyanide disposal. The table shows that cyanide removal by sodium hypochlorite with sodium hydroxide is also effective. The removal efficiency of cyanide gas with two spray towers was almost 100%. After the first spray tower, HCN concentration decreased from about $(240 \pm 50) \text{ mg/m}^3$ to $(35 \pm 15) \text{ mg/m}^3$. After the second spray tower, effluent HCN concentration was $(41 \pm 16) \text{ mg/m}^3$ to $(170 \pm 35) \text{ mg/m}^3$ after the first absorption stage and to $(183 \pm 37) \text{ mg/m}^3$ after the second stage, indicating that ammonia is one of the detected end products of cyanide degradation. Similar results are reported by Kao et al., who showed that *Klebsiella oxytoca* can biodegrade cyanide to non-toxic end products such as ammonia.

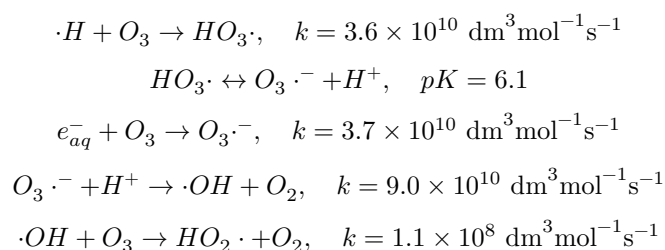
3.2.2 Wastewater Treatment System

Effects of initial concentrations: Pilot experiments were carried out to study variations in initial cyanide concentration at pH 9.8. Changes in cyanide concentration as a function of irradiation dose are presented in [Figure 9: see original paper] and [Figure 10: see original paper]. The degradation trend is consistent with bench-scale experiments. As seen in [Figure 9: see original paper], cyanide was removed more rapidly at lower initial CN^- concentration than at higher concentrations. At an irradiation dose of 1.5 kGy, removal efficiencies were 59.25%, 42.17%, and 33.88%, corresponding to initial CN^- concentrations of 21.08 mg/L, 55.63 mg/L, and 117.49 mg/L, respectively. Mudliar et al. reported that advanced oxidation processes such as UV light, hydrogen peroxide, and ozone are feasible options for treating cyanide-containing industrial wastewater. [Figure 9: see original paper] and [Figure 10: see original paper] show that electron beam irradiation is also a feasible method to achieve complete cyanide removal from wastewater at acceptable doses. When initial CN^- concentrations were 21.08 mg/L and 55.63 mg/L, irradiation doses of 6 kGy and 9 kGy were required, respectively, to meet national discharge standards. For higher initial CN^- concentrations such as 117.49 mg/L and 212.89 mg/L, higher irradiation doses of 15 kGy and 27 kGy were needed.

Effects of the initial solution pH: To study pH effects on electron beam radiolysis of cyanide solution, experiments were performed with an initial CN^- concentration of 55.63 mg/L. Wastewater pH was adjusted to 8.51, 9.88, and 12.07. Results are presented in [Figure 11: see original paper]. CN^- concentration decreased with increasing irradiation dose, but degradation efficiency was almost the same at pH 8.51 and 9.88. For example, at 1.5 kGy, CN^- removal efficiency was 40.39% and 42.17%, respectively, but only 29.59% at pH 12.07. At 10.5 kGy, CN^- concentration decreased to 0.67 mg/L, 0.86 mg/L, and 2.13

mg/L at pH 8.51, 9.88, and 12.07, respectively. The former two concentrations met national emission standards, while the third exceeded the standard by 113%. Since $\cdot\text{OH}$ radicals may be the main attacking species as discussed above, a possible cause for these results would be a decrease in $\cdot\text{OH}$ radical concentration at $\text{pH} > 11$, as shown in reactions (14)–(18).

Effects of O_3 , EB, and EB+ O_3 : It is generally thought that O_3 can increase the effectiveness of ionizing irradiation because it leads to increased oxidizing species concentration, as shown in reactions (14)–(18). The synergetic effect of O_3 on radiolytic decomposition of refractory compounds has been reported previously. To investigate O_3 effects on cyanide degradation, experiments were performed at 1.5 kGy, pH 9.56, and O_3 concentration of 2 mg/L. Results are shown in [Figure 12: see original paper].



For O_3 alone, CN^- removal efficiency was 27.14%, 22.77%, and 18.22%, respectively. For EB alone, CN^- removal efficiency was 59.25%, 42.17%, and 33.88%, respectively. However, CN^- removal significantly increased to 82.62%, 59.92%, and 48.19%, respectively, for EB+ O_3 . The extent of cyanide degradation by EB+ O_3 approximately corresponded to the sum of the decay with EB and with O_3 . Similar results are reported for catechol degradation. To achieve the same effect by EB alone, the required irradiation dose would need to increase from 1.5 kGy to 3 kGy, meaning the processing capacity of EB irradiation is doubled with 2 mg/L O_3 concentration.

Continuous stability experiment: Stability experiments were conducted for one week using a water regulating tank, where influent CN^- concentration was controlled at $(15 \pm 2) \text{ mg/L}$ and $\text{pH} 9-9.5$. The irradiation dose was 12 kGy throughout the process. [Figure 13: see original paper] show that outlet CN^- concentrations were below 0.5 mg/L after EB irradiation, meeting discharge standards. Considering previous work by Wang et al. investigating factors influencing radiation-induced degradation of sodium cyanide by high-energy electron beam, we may safely conclude that it is feasible to remove cyanide from wastewater by EB irradiation.

4 Conclusion

Based on results from bench-scale and pilot plant experiments, the following conclusions are outlined:

Cyanide was removed more rapidly at lower initial concentrations than at higher concentrations under both γ - and EB-irradiation. The radiation-induced decomposition of cyanide followed pseudo-first-order kinetics over the applied initial concentration range.

Water solution characteristics, such as composition and pH, significantly affected cyanide decomposition. These observations support that cyanide decomposition was greatly initiated by $\cdot\text{OH}$ radicals, while direct $e\text{q}^-$ attack on cyanide plays a minor role.

Removal efficiency of cyanide was higher in aerated than in de-aerated and static systems. Ozone increased the effectiveness of ionizing irradiation. The processing capacity of EB-irradiation was doubled with 2 mg/L O_3 concentration.

Stability experiments demonstrated that it is feasible to treat waste gas containing cyanide by combined chemical absorption and radiation degradation. During absorption and irradiation, cyanate and ammonia were found to be the main nitrogen-containing byproducts of cyanide degradation.

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