Comparative characterization of cyanide-containing steel industrial wastewater

Amit Mondal, Supriya Sarkar and Udayabhanu G. Nair

ABSTRACT

In the steel industry, cyanide in the wastewater is a major environmental concern. There are several chemical, physical, and biological treatment processes available for the removal of cyanide from industrial wastewater. But the efficacy of every treatment process depends on the complex elemental matrix of wastewater and the interference associated with them. Thus, water characterization plays a vital part in finding a suitable cyanide treatment process for any wastewater. Characterization data can give a clear overview of the complexity of cyanide in the wastewater, which ultimately helps in selecting the right remediation process. The present work includes comparative characterization of coke plant and blast furnace wastewater collected from an integrated steel plant. Three months of data for physico-chemical properties of the two different sources were analysed and compared. Pearson's correlation analysis of physico-chemical properties with free cyanide was also studied. The different forms of cyanide in coke plant and blast furnace water were also characterised, along with interference associated with them. It was observed that the water matrix of coke plant and blast furnace effluents are totally different. It was also evident that free cyanide concentration is much more affected in coke plant wastewater than in blast furnace water.

Key words | blast furnace, coke plant, cyanide, interferences, wastewater

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HIGHLIGHTS

- Interference effect is mainly observed in free cyanide content than weak acid dissociable cyanide content or strong acid dissociable cyanide content.
- Concentration of weak acid dissociable cyanide is significantly lower in coke plant water and blast furnace water than other types of cyanide present free cyanide and strong acid dissociable cyanide.
- Free cyanide concentration more affected in coke plant water due to the presence of more interfering elements than in blast furnace water.

INTRODUCTION

India is the second largest producer of crude steel in the world. More than 2% of India's GDP is from the iron and steel industry (Colla *et al.* 2017). Due to the many complex processes involved, it has become one of the most energy intensive sectors. In India, a relatively large quantity of water is consumed to produce steel (Alcamisi *et al.* 2014). Steel plants use a tremendous amount of water for different processes including waste transfer, cooling and dust control. The average consumption of water in the Indian steel industry is 5 m^3/ton of steel produced (Madhuri *et al.* 2017). After the completion of the processes, the wastewater produced contains different pollutants like phenol, ammonia, cyanide, chloride and several other toxic substances (Ghosh 2002). Therefore, treatment

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of the wastewater is essential, before discharging into the environment, to reduce the harmful elements below permissible limits. Several treatment methodologies have been designed and developed by researchers across the globe to meet the norms set by various governments and environment protection agencies (Alcamisi *et al.* 2014; Satyendra 2015). Among pollutants, cyanide is an extremely dangerous component due to its lethal effect on aquatic and human life (Halet *et al.* 2015; Singh *et al.* 2016). The wastewater from coke plants and blast furnaces has been identified as the main contributors of toxic aqueous cyanide from the iron and steel industries (Saha *et al.* 2018).

In coke plants, removal of coal derivatives and coke oven gas processing produce highly contaminated liquor containing tar, naphthalene, ammonia and other toxic components (Kwiecińska *et al.* 2017). After the recovery of by-products, the ammonia-still process is used for the removal of huge quantities of ammonia from the water (Park *et al.* 2008). Water from the by-product recovery process contains various toxic organic and inorganic compounds, such as ammonia, thiocyanate, phenols and cyanides. Wastewater generated in the gas cleaning stage is the main source of cyanide in coke plant water. In the biological oxygen treatment (BOT) plant, activated sludge is used for the microbial reduction of the cyanide, thiocyanate, ammonia and phenol content. After biological treatment, chemical treatment is used to reduce the cyanide value below the maximum contaminant limit (MCL) or permissible limit of 0.2 ppm. Large quantities of water (120 m^3/h) are discharged into the environment after the removal of colour through proper treatment.

Cyanide formation in blast furnaces mainly occurs through sodium and potassium present in raw materials (coke, ore, fluxes) as oxides, carbonates and silicates. Vaporized alkali metals react with nitrogen from the air blast and carbon from the coke to form alkali cyanide at temperatures of more than 1,000 °C (Petelin *et al.* 2008). This alkali cyanide is carried upward by top gas and leaves the blast furnace. During top gas purification and subsequent scrubbing of sludge, these alkali cyanides dissolve in the water. The equation for cyanide formation is as follows (M = Na, K).

$$M_2SiO_3 + 3C = 2M + Si + 3CO \quad (1)$$

$$M + 2C + N_2 = 2MCN \quad (2)$$

The scrubber water contains different forms of cyanide, suspended matter and appreciable amounts of chloride, ammonia and other dissolved materials. The recirculation of scrubber water results in a significant increase in the concentration of chloride along with cyanide and other dissolved contaminants in the blow down water. During the discharge of blast furnace blow down water, the cyanide enters the environment if not treated properly.

However, the characteristics of cyanide are significantly different in blast furnace and coke plant wastewater due to the differences in matrices. Cyanide present in the coke plant discharge water is associated with contaminants like ammonia, thiocyanate, sulphides, phenol and poly aromatic hydrocarbons (PAH). Whereas blast furnace blow down water contains cyanide along with chloride, ammonia, dissolved solids and other inorganic contaminants.

Blast furnace and coke plant wastewaters are mixtures of different types interfering elements, which interfere during cyanide remediation processes (Matino & Colla 2017). The treatment efficiency of these wastewaters is affected largely by the presence of different combinations of interfering elements such as thiocyanate, ammonia, phenol etc. (Dash *et al.* 2009; Mishra *et al.* 2018). Remediation processes respond differently to different types of cyanide complexes present in the wastewater matrix (Dash *et al.* 2009). Different cyanide measurement methods also encounter different types of interference due to these differences in the wastewater matrix (Biswas 2013; Pal & Kumar 2014).

Cyanide (CN^-) is a monovalent anion in which equimolar amounts of nitrogen and carbon atoms form triple covalent bonds. The chemical composition of cyanide in a solution depends on various factors such as pH, the presence of trace elements and other interfering elements (Jaszczak *et al.* 2017). Subsequently their environmental effects are also different. Cyanide is categorized based on the relative stability of its compounds and complexes in water as follows:

- Free cyanide (CN_F): the sum of hydrogen cyanide (HCN) and cyanide ions (CN^-). It exists as HCN at pH < 7 and as CN^- at pH > 10.5. It is the most toxic form cyanide. In water, CN_F is approximately a thousand times more toxic to aquatic organisms than to humans (Ikuta *et al.* 1999).
- Weak acid dissociable cyanide (CN_{WAD}): CN_{WAD} refers to weak to moderately strong metal cyanide complexes

which release cyanide ions in mildly acidic conditions. This includes complex cyanides of Zn, Cu, Cd, Hg, Ni and Ag that dissociate under pH 3–6.

- Strong acid dissociable cyanide (CN_{SAD}): CN_{SAD} are the strong metal cyanide complexes of Fe and Co, which require strong acidic conditions to dissociate and liberate hydrogen cyanide gas.

Total cyanide (CN_T) is the combination of all three types of cyanide (Figure 1). Toxicity of cyanide species follow the order of $CN_F > CN_{WAD} > CN_{SAD.}$ The legislation of the Indian government for cyanide discharge deals only with CN_F as it is highly toxic to living organisms and even deadly in nature. As per the Central Pollution Control Board (CPCB) of India, the discharge limit of CN_F to the environment is 0.2 ppm (Mondal *et al.* 2019).

Cyanide treatment in wastewater includes all three types of cyanide. The sensitivity of the treatment process is very much dependent on the characteristics of the different types of cyanide (Dash *et al.* 2009). The presence of thiocyanate in the wastewater can also create analytical interference during sample preservation with NaOH (Delaney *et al.* 2007; Sebroski 2011). To increase the accessibility of fresh water, reuse or recycling of wastewater is of utmost importance with suitable cyanide treatment technology. Detailed characterization of the wastewater matrix from different sources is thus essential to enable proper remediation methods with optimum efficiency.

The present study was carried out to find the concentration of different parameters of coke oven and blast furnace wastewaters that can affect cyanide concentration and its remediation. The wastewater matrix from these two sources are completely different as the sources of cyanide are different. In this study, the correlation of cyanide with different parameters has been analysed for these two different water matrices using Pearson correlation coefficient (r). In addition, the study includes the identification of the types of cyanide present in coke oven and blast furnace water and the underlying interference associated with them.

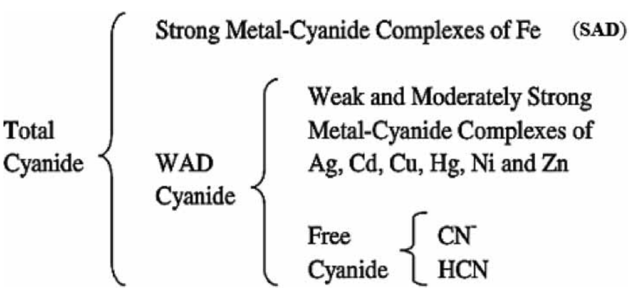

Figure 1 | Nature of Cyanide.

MATERIALS AND METHODS

Sampling of water

For the present experimental studies, water samples were collected from the blast furnace and coke oven of an integrated steel plant situated in the eastern part of India.

Analysis of blast furnace and coke oven water quality

Wastewater samples were collected in plastic jars from the blast furnace blow down and the coke plant for a period of 3 months with a sampling frequency of once every 3 days. Analysis of physico-chemical properties such as pH, total dissolved solids (TDS), biological oxygen demand (BOD), chemical oxygen demand (COD), contaminants such as ammonia, sulphate, thiocyanate, phenol, chloride, CN_F, complex cyanide (metal cyanide) and other cations and anions in the wastewater was carried out. The colour of the blast furnace and coke plant wastewater was also determined. Chemical characterization tests for all the parameters were performed in duplicate, other than CN_F analysis, which was conducted in triplicate.

Sample preservation and storage

For the analysis of cyanide, sample storage and preservation are very important steps. Cyanide volatilizes quickly at neutral pH, thereby losing the CN^- in the sample prior to its measurement. To increase the holding time of cyanide, sodium hydroxide (NaOH) was added immediately after collection of the wastewater samples to adjust the pH to >12. With this addition of NaOH, the specified holding time of cyanide is increased to 14 days (Ma & Purnendu 2010; Sebroski 2011).

Analysis of pH

The pH of the wastewater was measured using a pH meter (Systronics, India, digital pH meter, model no: 335).

Analysis of TDS

TDS is a measure of the combined content of all inorganic and organic substances contained in a liquid in molecular, ionized or micro-granular (colloidal sol) suspended form (Hussain 2019). TDS was measured using a TDS meter (Systronics, India, model no: 308), with the value reported in ppm.

Analysis of conductivity

Conductivity of the wastewater of these two sources were measured using a conductivity meter (Systronics, India).

Analysis of free cyanide (CN_F)

Analysis of CN_F was done potentiometrically using a cyanide ion selective electrode (ISE) (Thermo Fisher Scientific). Due to the low sample analysis time, approximately 5 min, many tests can be done at a time by this method. This method can be used to analyse CN_F in the range of 0.05–10 ppm.

Analysis of total cyanide (CN_T)

Total cyanide was measured by the total distillation method followed by colorimetric estimation using a spectrophotometer according to the procedure set out in APHA (2005). In the colorimetric method, CN^- in the alkaline distillate obtained from preliminary distillation is converted to CNCl by reaction with chloramine-T at $pH < 8$. After the reaction is complete, CNCl forms a red-blue colour on addition of pyridine-barbituric acid reagent.

Analysis of weak acid dissociable cyanide (CN_{WAD})

The analysis was done by distillation with weak acid at pH 4.5 followed by colorimetric analysis using a spectrophotometer according to the procedure set out in APHA (2005).

Determination of other elements in the wastewater

Blast furnace blow down water and coke plant water contain other interferences, including ammonia, sulphate, thiocyanate, phenol, nitrate, nitrite, iron, calcium, magnesium, etc. The analysis of cations such as Fe, Ca, Mg, Na, K, Zn, Cu, Cd and Ni was carried out in inductively coupled plasma optical emission spectroscopy (ICP-OES; Spectro Arcos). Whereas anions (sulphate, chloride, phosphate) were determined by ion chromatography (IC) (Metrohm). The phenol and total nitrogen content were measured by the Kjeldahl method using UDK 149 Automatic Distillation Unit with Titrator Connection (VELP Scientifica).

Determination of wastewater colour

Coke plant effluent after BOT treatment becomes a more intense dark brown colour due to the presence of degraded phenol compounds (Mijangos *et al.* 2006). However, this wastewater also contains relatively large quantities of various cyanide compounds, both complex and simple, and complex organic compounds, which are the major environmental concern if discharged or reused without any further treatment. It is, therefore, necessary to reduce the colour in the wastewater to a level that is not harmful to plant, animal or human life prior to disposal. The colour of the wastewater was measured by the colour instrument (Lovibond) and expressed in Pt/Co unit.

RESULTS AND DISCUSSION

CN_F in blast furnace and coke plant wastewater

CN_F in blast furnace water (BFW) and coke plant water (CPW) were analysed continuously for 3 months (Figure 2). Both the CPW and the BFW showed nearly similar (median) values of CN_F content: 5 ppm and 4.78 ppm respectively. But the variation of CN_F is higher in CPW, with a minimum of 2.8 ppm to a maximum of 9 ppm, compared to BFW where it varies from 3.03 ppm to 7.67 ppm.

Characterization of steel wastewater from two different sources

Steel wastewater from the blast furnace and coke plant were analysed for pH, TDS, colour, and the concentration of chloride and CN_F. Other important parameters such as sulphate, nitrate, nitrite, thiocyanate, phenol was also measured to assess their interfering effect with cyanide. In addition to this, other important parameters such as Na, K, Ca, Mg, COD, BOD, total Kjeldahl nitrogen (TKN) were also analysed to find the comparative characteristics of the two different sources of water. In total, 3 months of data were compiled. From the experimental results, it was observed that the wastewater characteristics are totally different in the two sources. Table 1 illustrates the concentration of the different parameters.

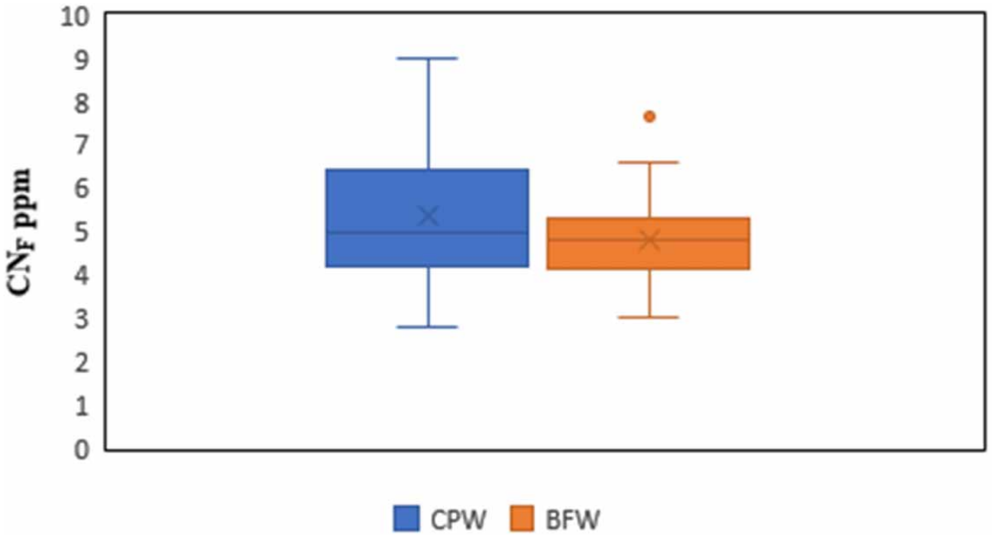

Figure 2 | Variation of CN_F in CPW and BFW.

The results showed that there is a large difference in colour between the two different water matrices, with average values of 2,795 Pt/Co and 6.23 Pt/Co for CPW and BFW respectively. In CPW, the colour of the water is dark brown, whereas in BFW it is nearly colourless. This is due to the formation of intensely coloured aromatic compounds (ortho- and para- benzoquinone) by catechol, resorcinol and hydroxyquinone, which formed during the degradation of phenol in BOT treatment (Mijangos *et al.* 2006).

The conductivity of CPW is lower than that of BFW, with average values of 4,978 ms and 8,359 ms respectively. This difference in conductivity is in accordance with the TDS value of the two sources: 2,782 ppt and 6,445 ppt for CPW and BFW respectively. A strong correlation between TDS and conductivity is in line with the research work by Choo-in (2019).

Average chloride concentration is high in both the sources, but the range is higher in BFW (923–2,094 ppm) than in CPW (894–1,451 ppm) (Colla *et al.* 2017). There is a huge difference in sulphate data between the two sources. The concentration of sulphate is very high in CPW (1,090–2,288) whereas in BFW it is very low (29–105) (Saha *et al.* 2018). Another major difference observed is in phenol and thiocyanate concentrations. Both are negligible in BFW, whereas the concentration is higher in CPW. The high phenol content is due to the decomposition of organic matter during coke making and subsequent release with CPW (Halet *et al.* 2015). COD and BOD values are higher in CPW and vary from 1,560 ppm to 2,164 ppm and 1,022 ppm to 1,242 ppm respectively, whereas BFW contains very low COD and BOD. Biological treatment is used in CPW to reduce this high load of BOD and COD (Mukherjee *et al.* 2012). TKN and total concentration of organic and ammoniacal nitrogen provide the information about the organic nitrogen content present in wastewater. Higher value of TKN in CPW may be due to the presence of higher thiocyanate and ammonia content. Concentration of Na in CPW and BFW is 1,164 and 300 ppm respectively. The high value in CPW is due to the use of sodium hydroxide in coke oven gas cleaning and is a by-product of the recovery process (Wang *et al.* 2002; Park *et al.* 2008).

Pearson's correlation analysis between CN_F and different physico-chemical properties

Pearson's correlation coefficient (r) is significantly different in CPW and BFW. Parameters such as pH, sulphate ($S0_4^{2-}$), Na and K show moderate correlation with CN_F in BFW with r values of 0.55, −0.52, 0.4 and 0.41 respectively. In CPW, CN_F is moderately correlated with thiocyanate and conductivity, with r values of 0.45 and −0.46 respectively. No strong correlation of CN_F exists with any single physico-chemical parameter. Other than some moderate correlations, most of the correlations are weak to very weak in both the sources of water. In addition, no strong correlation of CN_F with the major interfering elements such as thiocyanate, phenol, sulphate, TKN, etc. exist.

Table 1 | Three-month average characterization data of BFW and CPW

		CPW				BFW			
Parameter	**Unit**	**Average**	**Min**	**Max**	**SD***	**Average**	**Min**	**Max**	**SD***
CN_F	ppm	5.34	2.8	9.00	1.63	4.78	3.03	7.76	0.98
Colour	Pt/Co	2,795	2,550	2,950	94.94	6.23	0.10	64.8	17.82
pH	–	8.73	8.10	9.40	0.29	7.96	7.14	8.98	0.53
Conductivity	mS	4,978	4,170	5,710	395	8,359	7,823	8,740	262
TDS	ppt	2,782	2,231	3,350	254	6,445	5,905	6,934	217
Cl	ppm	1,196	894	1,451	124	1,218	923	2,094	188
Fe	ppm	4.20	0.50	9.40	1.71	0.09	0.03	1.10	0.18
SO_4^{2-}	ppm	1,330	1,090	2,288	225	54.34	29	105	15.98
Na	ppm	1,164	880	1,355	137	300	75	566	83.84
K	ppm	695	50	1,289	420	454	302	622	62.59
Ca	ppm	73.20	45.20	105	12.80	175	73	311	60.75
Mg	ppm	14.19	7.00	23.30	4.00	48.05	40	67	5.88
Total P	ppm	113	0.24	644	142	0.14	0.01	1.01	0.25
COD	ppm	1,931	1,560	2,164	79.03	53.89	12.00	102.00	25.34
BOD_5	ppm	1,242	1,022	1,446	42.30	30.92	15.60	42.30	6.80
Thiocyanate	ppm	423	301	542	71.63	7.14	1.02	15.67	4.16
Phenol	ppm	557	414	662	60.97	0.02	0.01	0.10	0.03
NO_3	ppm	18.43	9.80	71.10	15.31	18.80	16.42	23.00	1.50
NO_2	ppm	3.95	2.54	5.65	1.58	0.42	0.04	2.10	0.38
TKN	ppm	151	83	252	37.84	90.85	71.30	109	7.87

*SD, standard deviation.

Types of cyanide in different sources of water

The average results of different types of cyanides in CPW and BFW are presented in Figure 3. It was observed that the concentration of CN_{WAD} is lower than CN_F and CN_{SAD} in both the sources of water, which may be due to the absence of a significant amount of CN_{WAD} forming cations such as Zn, Cu, Cd and Ni, as shown in Table 2.

Finding the effect of known interferences in cyanide solution

Salts of interfering elements were added one by one to 2 ppm standard cyanide solution to study the effect of interference.

Cyanide concentration was positively biased with the addition of sulphide, whereas negative bias was observed in presence of thiocyanate salt (Table 3). No significant impact was observed on cyanide by the addition of ammonia and phenol. In addition to this, nitrate salt had small negative impact on the cyanide value. Many cyanide treatment methods pass through the formation of thiocyanate. Stability of thiocyanate is greater than that of cyanide, which makes its treatment difficult (Gould *et al.* 2012). So, elimination of thiocyanate interference is of the utmost important for cyanide analysis and cyanide treatment.

Finding the interference effect in BFW

Experiments were carried out to find the effect of interference on the concentration of CN_F in BFW. Figure 4 shows that there is no significant impact of the addition of ammonium, thiocyanate and sulphide salts on all types of cyanide in BFW. Only nitrate creates a negative bias in the concentration of CN_F and CN_T. The addition of phenol also has no significant impact on any types of cyanide. During this interference study, it was evident that the

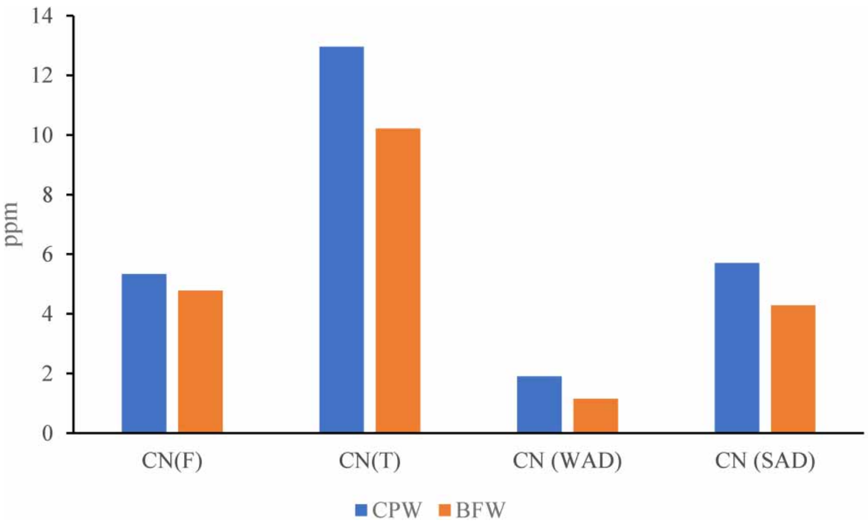

Figure 3 | Comparison of CN_F, CN_{WAD}, CN_{SAD} and CN_T in two different sources of water.

Table 2 | Zn, Cu, Cd and Ni in CPW and BFW

Parameter	CPW	BFW
Zn (ppm)	1.53	< 0.1
Cu (ppm)	< 0.1	< 0.1
Cd (ppm)	< 0.1	< 0.1
Ni (ppm)	< 0.1	< 0.1

Table 3 | Variation of CN_F in presence of know interferences in standard solution

Addition of known interferences	Initial CN_F (ppm)	After treatment, CN_F (ppm)
Ammonium salt	2.0	2.1
Nitrate salt	2.0	1.7 ↓
Thiocyanate salt	2.0	1.2 ↓
Sulphide salt	2.0	4.2 ↑
Phenol	2.0	2.05

interference effect is significant mainly for the CN_F content rather than the CN_{WAD} or CN_{SAD}.

Finding the interferences effect in CPW

Figure 5 illustrates the interference effect results in CPW, which show that addition of thiocyanate and sulphide has a great impact on CN_F as well as total cyanide. The addition of nitrate and ammonium has a small impact on CN_F only. No significant changes were observed in any type of cyanide after the addition of phenol to CPW. The interference effect of all these elements is only evident in free and total cyanide, which is similar in BFW. No significant effect was observed on the CN_{SAD} and CN_{WAD} values.

SUMMARY AND CONCLUSION

The physico-chemical properties of real coke plant and blast furnace wastewater collected from a steel plant were analysed and compared. CPW is associated with very high amounts of phenol, sulphate, thiocyanate, COD, BOD and colour compared to BFW. Whereas TDS, conductivity, Ca and Mg are higher in BFW. No strong correlation exists between CN_F and other physico-chemical properties of CPW and BFW, as shown by Pearson's correlation analysis. Variation in CN_F content is higher in CPW than BFW, with standard deviations of 1.63 and 0.98 respectively. The interference effect is mainly observed in CN_F content, as CN_{WAD} and CN_{SAD} do not shown any significant changes with the addition of phenol, nitrite, ammonium, thiocyanate and sulphate salts. The concentration of CN_{WAD} is significantly lower (1.91 ppm and 1.15 ppm in CPW and BFW respectively) than other type of cyanide present. CN_F concentration is affected more in CPW due to the presence of more interfering elements than in BFW. The results showed that the water matrix is completely different in the

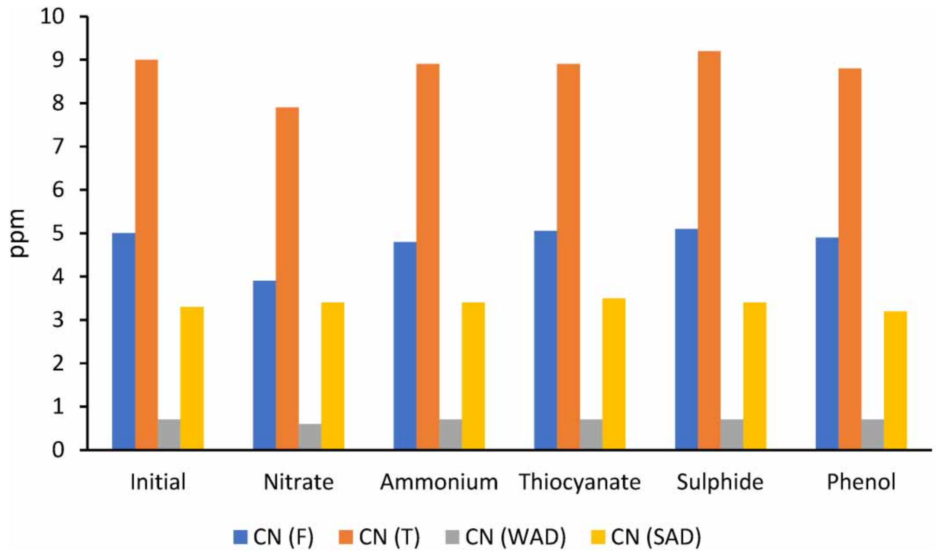

Figure 4 | Variation of cyanide concentration in presence of known interferences in BFW.

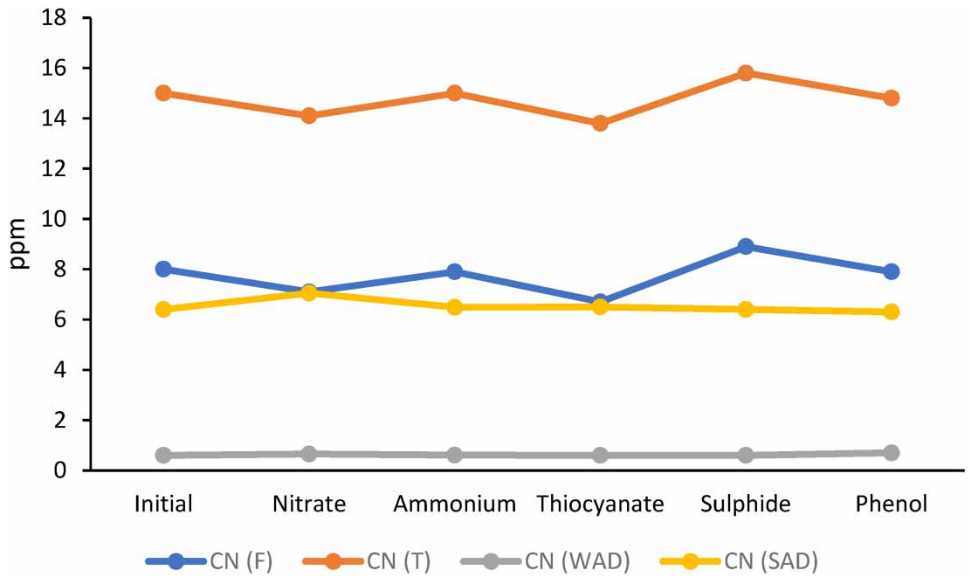

Figure 5 | Variation of cyanide concentration in presence of known interferences in CPW.

two sources of water. Different treatment processes for the two different sources of wastewater are therefore recommended for disposal or reuse.

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DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

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