Selective Separation of Fe (III) & Cr (III) from acidic waste water

Jayraj R Padshala[1], Dr. Shina Gautam[2]
[1]*student of chemical engineering, SRICT, Ankleshwar*
[2]*Associate professor of chemical engineering, SRICT, Ankleshwar*

Abstract- **There are several acidic waste water from chemical process industries which consist of heavy metals. One of the waste water from process industries has been selected to seprate selectively iron and chromium from acidic wastewater. The concentration of both metals are in range of 500-600 ppm first precipitation of iron and chromium will be evaluated to reduce the concentration. After maximum removal reactive extraction will be applied to separate metals. Design of experiments will be applied for metals selectively. This waste water treatment has not found in literature as per our findings. It is applied first time as per methodology explained.**

Index Terms- **Chemical Precipitation mehod, acidic waste water, NoaH, pH meter, Reagents, Expriment setup, , etc.**

1. INTRODUCTION

Heavy metal pollution has become one of the most serious environmental problems today. The treatment of heavy metals is of special concern due to their recalcitrance and persistence in the environment. In recent years, various methods for heavy metal removal from wastewater have been extensively studied.

In this reviews the current methods that have been used to treat heavy metal wastewater and evaluates these techniques. These technologies include chemical precipitation, and another methods likes (ion-exchange, adsorption, membrane filtration, coagulation eflocculation, flotation and electrochemical). If the most frequently studied for the treatment of heavy metal wastewater.

Chemical precipitation is effective and by far the most widely used process industry because it is relatively simple and inexpensive to operate. In precipitation processes, chemicals react with heavy metal ions to form insoluble precipitates. The forming precipitates can be separated from the water by sedimentation or filtration. And the treated water is then decanted and appropriately discharged or reused. The conventional chemical precipitation processes include hydroxide precipitation and sulfide precipitation.

1.1 Separation of Fe(III) & Cr(III) From acidic waste water various methods uses

1. Chemical Precipitation
2. Ion-exchange
3. Adsorption
4. Membrane filtration
5. Coagulation
6. E-Flocculation
7. Flotation
8. Electrochemical
9. Solvent Extraction

1.2 Theory of Chemical Precipitation and Efficiency

Three theories have been profounded to explain the effects and efficiency of chemical precipitation.

The first theory uses the fact that heavy metal salts when treated with alkaline substances, form heavy voluminous precipitates which carry down true and colloidal suspensions by means of mechanical entrapment Salts of iron, aluminium and zinc fall into this classification.

The second theory is electronic in nature. It has been demonstrated that colloidal particles possess an electric charge. Since these charges are alike, the particles repel each other and thus tend to remain in suspension If a colloidal particle with an opposite charge is added, the charges neutralize and settling of the particles is effected. This explains the efficiency of the multivalent ions and why ferxir: salts are more efficient than ferrous salts. Clay suspensions are also claimed to exert, a charge neutralizing effect.

The third theory pertaining to the final theoretical effect is largely physical. Insoluble substances which have a large particle surface area can effectively absorb colloids ; they can also act as nuclei for the initiation of precipitation. Activated charcoal is a material with this type of action.

1.3 Hydroxide Precipitation

The most widely used chemical precipitation technique is hydroxide precipitation due to its relative simplicity, low cost and ease of pH control (Huisman et al., 2006). The solubilities of the various metal hydroxides are minimized in the pH range of 8.0 to 11.0. The metal hydroxides can be removed by flocculation and sedimentation.

A variety of hydroxides has been used to precipitate metals from wastewater, based on the low cost and ease of handling, lime is the preferred choice of base used in hydroxide precipitation at industrial settings (Baltpurvins et al., 1997) (Table 1). Hydroxide precipitation process using $Ca(OH)_2$ and NaOH in removing Cu(II) and Cr(VI) ions from wastewater was evaluated by Mirbagheri and Hosseini (2005).

The Cr(VI) was converted to Cr(III) using ferrous sulfate. Maximum precipitation of Cr(III) occurred at pH 8.7 with the addition of Ca(OH)2 and the concentration of chromate was reduced from 30 mg/L to 0.01 mg/L. The cupro-ammonia was reduced by aeration and the optimum pH for maximum copper precipitation was about 12.0 for both Ca(OH)2 and NaOH and the concentration of copper was reduced from 48.51 mg/L to 0.694 mg/L..

The concentrations of chromium, in effluents can be reduced from initial concentration of 100.0 mg/L to 0.08 mg/L. In hydroxide precipitation process, the addition of coagulants such as alum, iron salts, and organic polymers can enhance the removal of heavy metals from wastewater. Charerntanyarak (1999) employed chemical coagulation and precipitation by lime to treat synthetic wastewater consisting of Zn, Cd, Mn and Mg at the concentration of 450, 150, 1085 and 3154 mg/L, respectively.

He found that the optimum pH was more than 9.5 and the treated wastewater could meet the Wastewater Standard of the Ministry of Industry. Moreover, if coagulant was added, the residual concentration of heavy metal can be decreased further. Although widely used, hydroxide precipitation also has some limitations. Firstly, hydroxide precipitation generates large volumes of relatively low density sludge, which can present dewatering and disposal problems (Kongsricharoern and Polprasert,1995). Secondly, some metal hydroxides are amphoteric, and the mixed metals create a problem using hydroxide precipitation since the ideal pH for one metal may put another metal back into solution. Thirdly, when complexing agents are in the wastewater, they will inhibit metal hydroxide precipitation.

1.4 Sulfide Precipitation

Sulfide precipitation is also an effective process for the treatment of toxic heavy metals ions. One of the primary advantages of using sulfides is that the solubilities of the metal sulfide precipitates are dramatically lower than hydroxide precipitates and sulfide precipitates are not amphoteric. And hence, the sulfide precipitation process can achieve a high degree of metal removal over a broad pH range compared with hydroxide precipitation. Metal sulfide sludges also exhibit better thickening and dewatering characteristics than the corresponding metal hydroxide sludges. Özverdi and Erdem (2006) The mechanism governing the metal removal processes was determined as chemical precipitation at low pH (<3) due to H_2S generation and adsorption at high pH (in the range of 3 to 6)

Hydrogen sulfide reacts with divalent soluble metals to form insoluble metal sulphides Some attractive findings were reported by Kousi et al. (2007) who developed an upflow fixed-bed SRB to monitor for the treatment of zinc-bearing wastewater. They found that the reactor has a considerable capacity of completely reducing sulfates for initial concentrations up to 6000 mg/L, completely removing soluble zinc for initial concentrations up to 400 mg/L and completely removing TOC for initial concentrations up to 1500 mg/L. The possibility of using SRB for the treatment of an acid mine drainage was also studied (Alvarez et al., 2007). However, there are potential dangers in the use of sulfide precipitation process. As we know, heavy metal ions often in acid conditions and sulfide precipitants in acidic conditions can result in the evolution of toxicH2S fumes. It is essential that this precipitation process be performed in a neutral or basic medium. Moreover, metal sulfide precipitation tends to form colloidal precipitates that cause some separation problems in either settling or filtration processes.

1.5 Chemical precipitation combined with other methods

Chemical precipitation has been shown to be successful in combination with other methods. González-Muñoz et al. (2006) reported sulfide precipitation to reuse and recover heavy metal ions and employed nanofiltration as a second step. Results indicated sulfide precipitation was successful in reducing the metal content and nanofiltration yielded solutions capable to being directly reused in the plant. Ghosh et al. (in press) used electro-Fenton process and chemical precipitation to treat rayon industry wastewater to reduce its COD (2400 mg/L) and Zn2þ (32 mg/L). Results revealed that approximately 88% COD was reduced using electro-Fenton method and zinc removal (99 to 99.3%) was attained in the range of pH 9 to 10 using lime precipitation. There are some reports on chemical precipitation in combination with ion-exchange treatments. Papadopoulos et al. (2004) reported using ion-exchange processes individually and then combining with chemical precipitation in removing nickel from wastewater streams from a rinse bath of aluminum parts. They found that the individual application of ion exchange led to the removal of nickel up to 74.8%, while using the combination of ionexchange and precipitation processes, higher removal from 94.2% to 98.3% was obtained. Besides, treating acid mine water by the precipitation of heavy metals with lime and sulfides, followed by ion exchange was also reported (Feng et al., 2000).

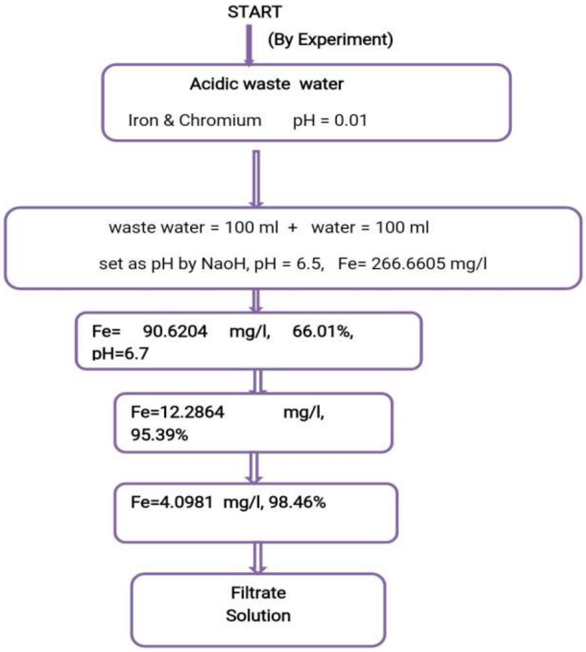
1.6 Different Reagents

Sr. No.	Reagent	Material
1	Na_2CO_3	Ores of Sb, As, Cr, Fe, Mo, V, Zr, sulphides, pyrites etc
2	Na_2O_2	Ores of Sb, As, Cr, Mo, Ni, V, Sn etc. in sulphide for sulphur determination
3	NaOH, KOH	Oxidized Sb ore, ores of Cr, Sn, Zn, Zr, sulphides and sulphates etc
4	$KHSO_4$, $K_2S_2O_7$	Ores of Al, Sb, Cu, Cr, Co, Fe, Mn, Ni, Rh, Ta, Ti, W etc
5	Na_2CO_3 & K_2CO_3	Silicates of Al, Fe, F, N, Se, Te, etc.
6	Na_2CO_3	Silicates of Al, Fe, Ca, Cr etc, halides of Ag, sulphates of Ba & Pb.

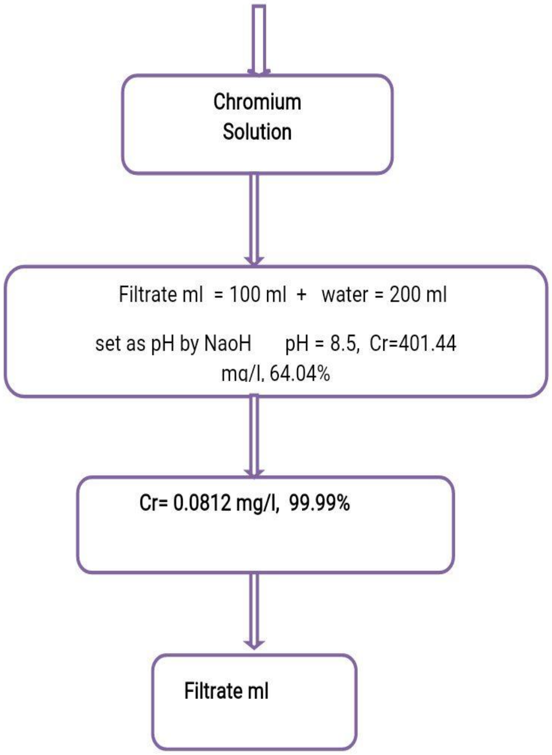
Table 1 Different Reagents

2 EXPERIMENT PROCEDURE

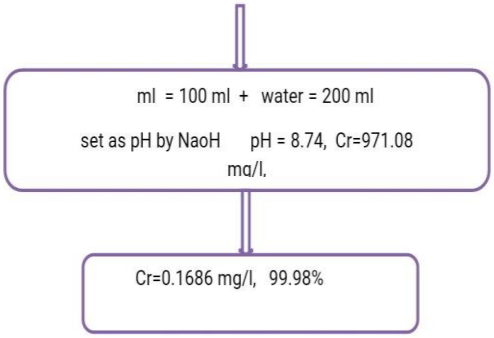
Part 1 Fe(III) Separation from acidic Waste water



Part 2 Cr(III) Separation from Fe(III) Filtrate after ml



Part 3 Cr(III) Separation from intial acidic waste water



- if three parts separation of Fe(III) & Cr(III) from acidic waste water.
- First of Fe(III) seprate after Cr(III) seprate .
- Sample Check for AAS

3 STEP WISE EXPERIMENT PROCEDURE

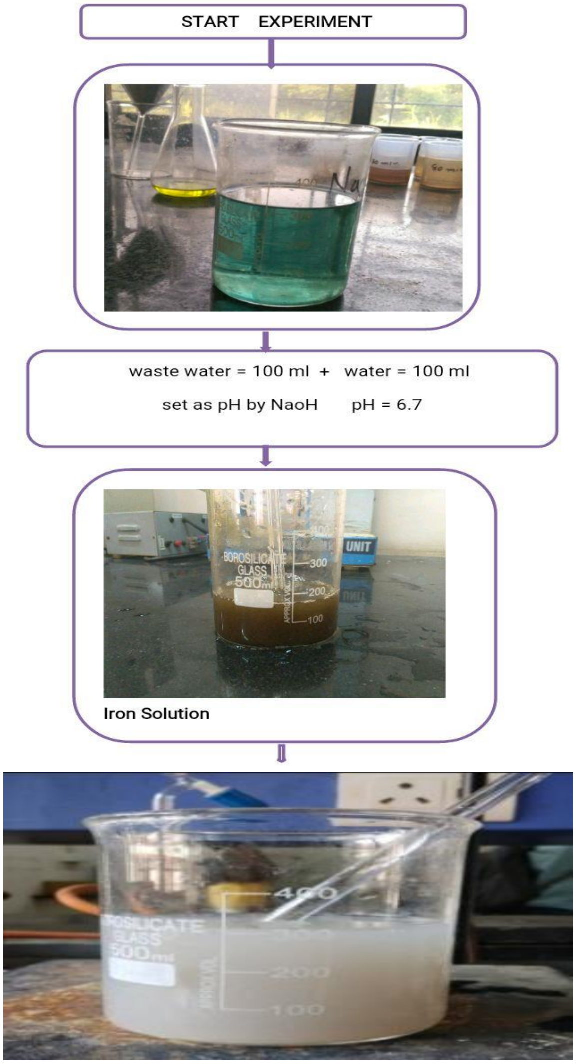


Fig.1 NaoH Solution

Fig.2 Iron Precipitate

Fig. 3 Filtration of Iron

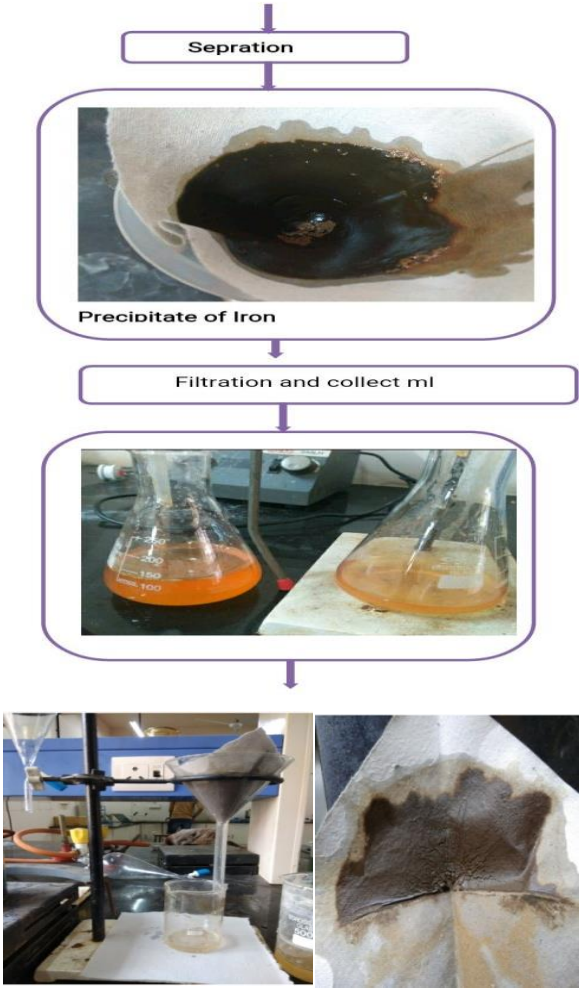


Fig.4 Filtration

Fig.5 Chromium Pricipitate Solution

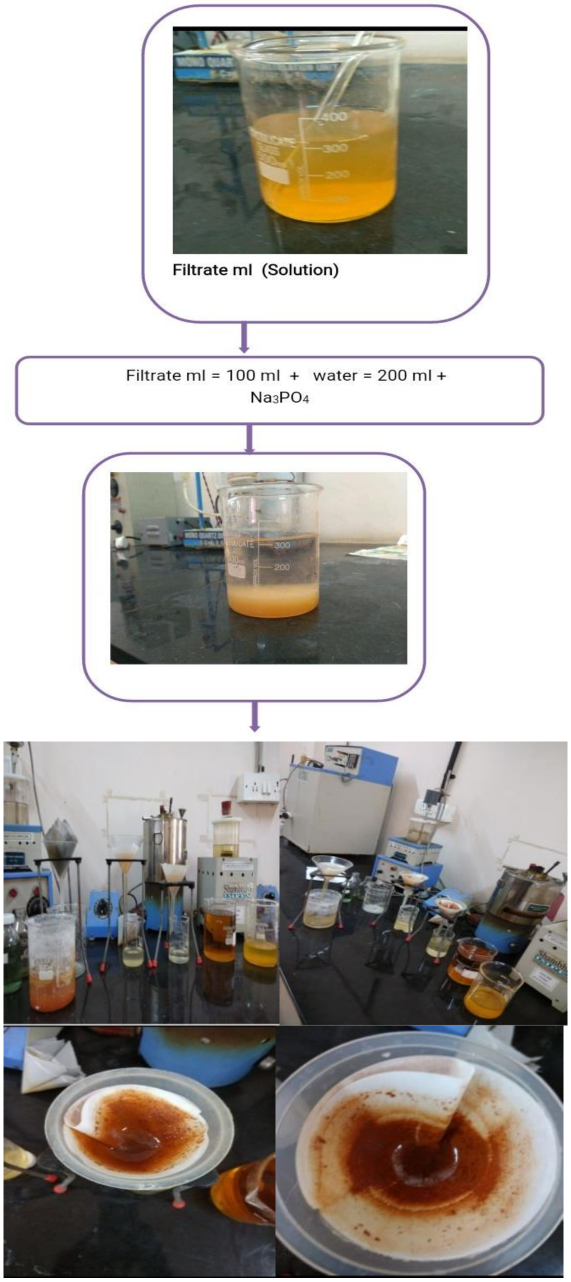
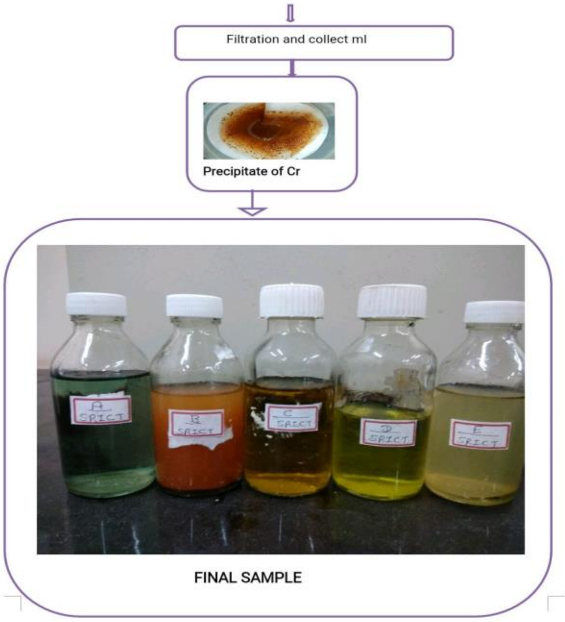


Fig. 6 Chromium Precipitate



4 RESULTS AND DISCUSSION

If the Selective saparation of Iron and Chromium from acidic waste water in two part seprate first of Fe(III) & Cr(III)

4.1Part (1) Fe(III) Separation

- Waste water = 100 ml
- Distilled water = 100 ml
- Ratio = 1 : 1
- NaOH= 18 gm
- pH of this mix. was adjusted dropwise with a solution of NaOH in distilled water.

Sr. No.	Waste Water	H_2O ml	NaOH gm	pH	Results
1	-------	----	----	0.01	Nil
2	100 ml	100	5	1	Nil
3	100 ml	100	4	2	Nil
4	100 ml	100	2	4	Nil
5	100 ml	100	2	4.5	Nil
6	100 ml	100	2	5	Nil
7	100 ml	100	1	5.8	Nil
8	100 ml	100	1	6	Nil
9	100 ml	100	1	6.5	Fe
10	100 ml	100	0.8	6.85	Fe

Table 2 Separation of Fe(III)

4.2 Part (2) Cr(III) Separation

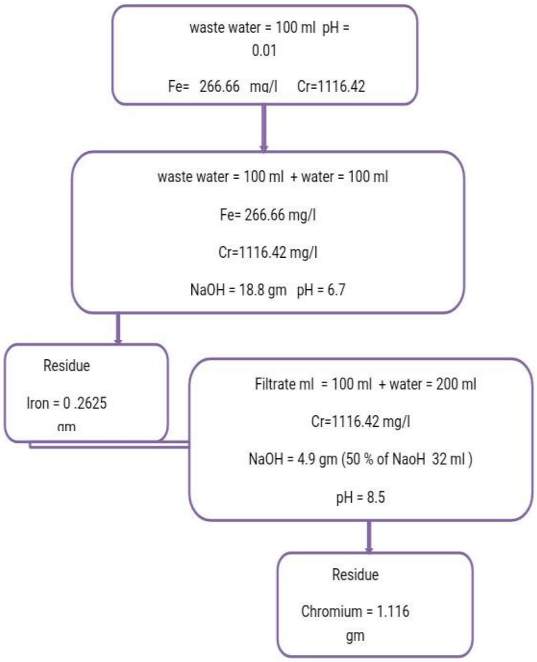
- Waste water = 100 ml
- Distilled water = 200 ml

- Ratio = 1 : 2
- Na_3PO_4= 1 gm
- pH of this mix. was adjusted dropwise with a 50 % solution of NaOH in distilled water

Sr. No.	Waste Water + H_2O + $Na_3PO_4.7H_2O$	50% NaOH Solution	pH	Results
1	100 ml + 200 ml --------	-----	6.7	Nil
2	100 ml + 200 ml 1 gm	5 ml	7	Nil
3	100 ml + 200 ml 1 gm	5 ml	7.3	Nil
4	100 ml + 200 ml 1 gm	5 ml	7.5	Nil
5	100 ml + 200 ml 1 gm	5 ml	7.8	Nil
6	100 ml + 200 ml 1 gm	4 ml	8	Nil
7	100 ml + 200 ml 1 gm	4 ml	8.1	Nil
8	100 ml + 200 ml 1 gm	2 ml	8.2	Nil
9	100 ml + 200 ml 1 gm	1 ml	8.4	+Ve
10	100 ml + 200 ml 1 gm	1 ml	8.5	+Ve

Table 3 Separation of Chromium(III)

4.3 Material balance



➢ Separation of Fe(III)

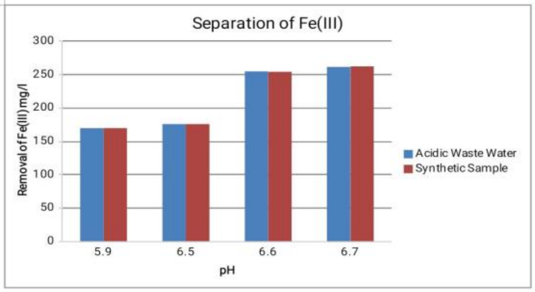


Fig. 7 Compare Pure & acidic waste water of Fe(III)

➢ Separation of Cr(III)

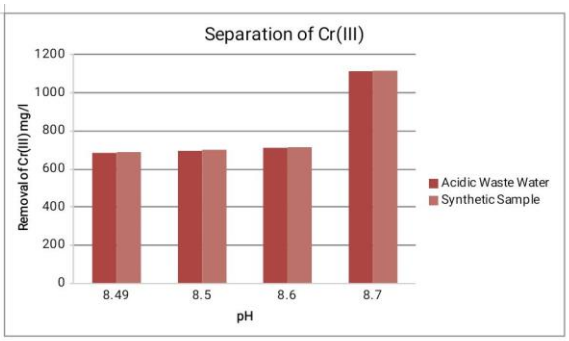


Fig. 8 Compare Pure & acidic waste water of Cr(III)

➢ Separation of Fe(III) & Cr(III)

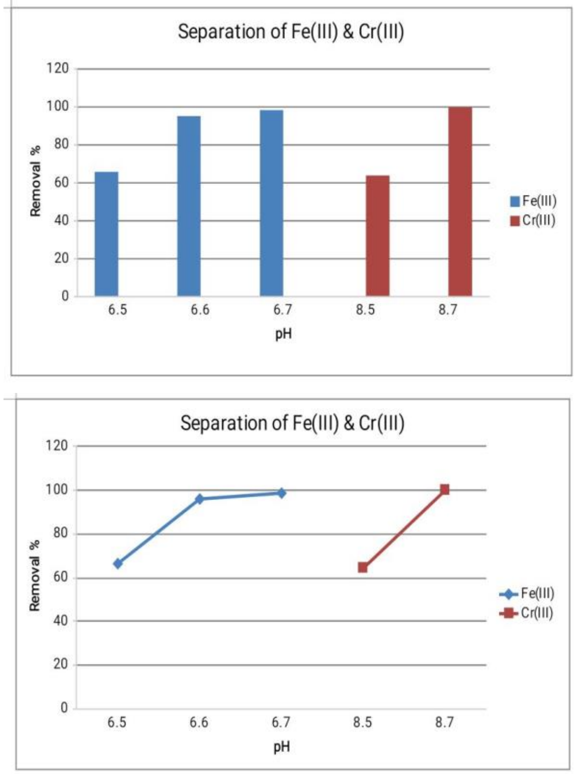


Fig. 9 Compare acidic waste water of Fe(III) & Cr(III)

5 CONCLUSIONS

Hazardous heavy metal pollution of wastewater is one of the most important environmental problems throughout the world. To meet the increased more and more stringent environmental regulations, a wide range of treatment technologies such as chemical precipitation, i have selective seprate of Fe(III) & Cr(III) from acidic waste water. and two part separation first part Iron after

Chromium seprate using chemical precipitation method. seprate of Chromium from (Filtrate ml Fe) acidic waste water

ACKNOWLEDGMENTS

I would like to thank Dr. Shina Gautam (Associate Professor, Chemical Engineering, Dept, SRICT) to support for this project. They have been an excellent advisors, providing sound technical guidance. This Work Was supported by the Department of chemical Engineering and Shroff S.R. Rotary Institute of Chemical Technology(SRICT), Vatariya, Ta.-Valia, Dist. – Bharuch..Last but not the least; I would like to thank Almighty God, my parents, and my friends for support and excellent co-operation to build my moral during the work.

REFERENCES

[1] Abo-Farha, S.A., Abdel-Aal, A.Y., Ashourb, I.A., Garamon, S.E., 2009. Removal of some heavy metal cations by synthetic resin purolite C100. J. Hazard. Mater. 169, 190-194.

[2] Agoubordea, L., Navia, R., 2009. Heavy metals retention capacity of a non-conventional sorbent developed from a mixture of industrial and agricultural wastes. J. Hazard. Mater. 167, 536-544.

[3] Ahmad, A.L., Ooi, B.S., 2010. A study on acid reclamation and copper recovery using low pressure nanofiltration membrane. Chem. Eng. J. 56, 257-263.

[4] Ahn, C.K., Park, D.,Woo, S.H., Park, J.M., 2009. Removal of cationic heavy metal from aqueous solution by activated carbon impregnated with anionic surfactants. J. Hazard. Mater. 164, 1130-1136.

[5] Ajjabi, L.C., Chouba, L., 2009. Biosorption of Cu2þ and Zn2þ from aqueous solutions by dried marine green macroalga Chaetomorpha linum. J. Environ. Manage. 90, 3485e3489.

[6] Amudaa, O.S., Adelowoa, F.E., Ologunde, M.O., 2009. Kinetics and equilibrium studies of adsorption of chromium(VI) ion from industrial wastewater using Chrysophyllum albidum (Sapotaceae) seed shells. Colloid Surf. 68, 184-192

[7] Medina, B.Y., Torem, M.L., de Mesquita, L.M.S., 2005. On the kinetics of precipitate flotation of Cr III using sodium dodecylsulfate and ethanol. Miner. Eng. 18, 225-231.

[8] B.R. Reddy, P.V.R. Bhaskara Sarma, Extraction of iron(III) at macro-level concentrations using TBP, MIBK and their mixtures, Hydrometallurgy 43 (1996) 299–306.

[9] C. Parija, B.R. Reddy, P.V.R. Bhaskara Sarma, Recovery of nickel from solutions containing ammonium sulphate using LIX 84-I, Hydrometallurgy 49 (1998) 255–261.

[10] C. Parija, P.V.R. Bhaskara Sarma, Separation of nickel and copper from ammoniacal solutions through co-extraction and selective stripping using LIX84 as the extractant, Hydrometallurgy 54 (2000) 195–204.

[11] A.I. Ali, J.A. Daoud, H.F. Aly, Recovery of copper from sulphate medium by LIX-84 in kerosene, J. Chem. Technol. Biotechnol. 67 (2) (1996) 137–142.

[12] J. Saji, T. Prasada Rao, C.S.P. Iyer, M.L.P. Reddy, Extraction of iron(III) from acidic chloride solutions by Cyanex 923, Hydrometallurgy 49 (1998) 289–296.

[13] Deep, P.F.M. Correia, J.M.R. Carvalho, Selective recoveries of Fe(III) and Cr(III) from a tannery filtrate using Cyanex 923, Anal. Chim. Acta 558 (2006) 254–260.

[14] E.I. El-Shafey, P.F.M. Correia, J.M.R. Carvalho, Sep. Sci. Technol. 40 (2005) 2297–2329.

[15] G.M. Ritcey, A.W. Ashbrook, Solvent extraction principles and applications to process metallurgy, vol. 2, Elsevier Science, Amsterdam, 1979.

[16] M.I. Stefanakis, A.J. Monhemius, Hydrometallurgy 19 (2) (1987) 187–198.

[17] J. Saji, M.L.P. Reddy, Hydrometallurgy 61 (2) (2001) 81–87.

[18] J. Jayachandran, P.M. Dhadke, Talanta 44 (7) (1997) 1285–1290.

[19] F. Principe, G.P. Demopoulos, Hydrometallurgy 74 (1) (2004) 93–102.

[20] G.P. Demopoulos, F.T. Principe, Proceedings of the EPD Congress, The Minerals, Metals and Materials Society, 1998, pp. 267–287.

[21] M.R.C. Ismael, J.M.R. Carvalho, Proc. Int. Solvent Extrac. Conf. 1 (1996) 599–604.

[22] A.I. Vogel, A Textbook of Quantitative Chemical Analysis, 5th edn., ELBS, Longman, England, 1989, p. 690.

[23] T. Sekine, Y. Hasegawa, Solvent Extraction Chemistry, Fundamentals and Applications, Marcel Dekker, New York, 1977, pp. 48–50.
[24] Ismael, M. R. C.; Carvalho, J. M. R. Extraction of iron(III) from aqueous sulphuric acid solutions using a packed column. Proc. Int. SolVent Extr. Conf. 1999, 2, 915-919.
[25] Miralles, N.; Sastre, A. M.; Figuerola, E.; Martinez, M. Solvent extraction of iron(III) by bis(2,4,4-trimethylpentyl) phosphinic acid: Experimental equilibrium study. Hydrometallurgy 1992, 31 (1-2), 1-12.