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Extraction of acid and iron values from sulphate waste pickle liquor of a steel industry by solvent extraction route

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Abstract

The extraction of sulphuric acid from the actual solutions generated during the process of pickling of steel from a local tube company dealing with the processing of iron and steel tubes using Alamine 336 has been studied in detail. Various parameters were optimised for the maximum extraction of acid such as concentration of organic extractant, time for equilibration, O/A ratio, acid concentration in the feed, temperature for extraction was varied from 30 to 60 °C. Stripping of loaded acid was done with distilled water at 60 °C. Stripping parameters studied were, temperature and time effect on equilibration, O/A ratio, multiple contact of the same strippant with fresh loaded organic. The effects of the numbers of extraction and stripping stages on the extraction and stripping of sulphuric acid recovery are discussed. After the extraction of acid from WPL, iron values in the raffinate were extracted with a binary solvent mixture consisting of MIBK and D2EHPA which shows a synergistic effect on iron extraction.
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1. Introduction

Pickle liquors from the primary metal and metal finishing industries comprise a major source of toxic industrial wastes. Pickling is a method used in sheet and wire mills or metal fabricating plants to remove oxide and scale from the surface of the metal sheet, strip, wire, or parts before another operation, such as galvanizing, electroplating, or painting, by passing the metal products through an acid bath. Steels are usually pickled in 15–20% HCL or H_2SO_4 at temperature up to 100 °C. During pickling the scale oxides dissolve to give iron(II) sulphates/chloride and repeated pickling in the same liquor lead to the generation of pickle liquor containing mineral acids along with various metals such as iron, Zn, Cr, Ni etc depending upon the type of steel treated. This pickle liquor then becomes unacceptable for further use and is bled off as spent pickle liquor. Due to the corrosive nature and presence of the high amount of dissolved iron (II) this WPL cannot be disposed off without pretreatment. In industrial processes, such spent pickling solutions are usually treated with lime to neutralise acid and the sludge generated is dumped. Alternatively SPL is evaporated and cooled to 5 °C, and then to 0 °C to crystallize iron(II) sulphate as its heptahydrate. To recover and reuse the acid and the dissolved iron(II) in the spent pickling solutions as valuable materials and to

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obtain an environmentally benign effluent, an alternative method for treating spent pickling solutions is essentially required. Studies have been reported on the use of Tri-butyl phosphate (TBP) for the extraction of acid (Petkovic et al., 1992; Ritcey and Ashbrook, 1984a,b; Cox, 1992; Eyal et al., 1993). The extraction of mineral acids by Cyanex 923 (Alguacil and Lopaz, 1996) was also investigated and reported. This study deals with the extraction of acid using Alamine 336 a tertiary amine. Similarly selective and quantitative separation of iron (III) from the waste pickle liquor can be effected through solvent extraction. A considerable amount of work has been reported (Demopoulos and Gefvert, 1984; Alguacil and Amer, 1986; Islam and Biswis, 1979; Chen et al., 1992; Sekine et al., 1976; Roddy et al., 1971; Alguacil et al., 1987; Sytefanakis and Monhemius, 1885) either on the removal of iron(III) as an impurity from leach solution or its extraction from dilute solutions. No systematic work has been reported on the solvent extraction of iron(III) from the concentrated sulphate solutions. Several solvents such as methyl isobutyl ketone (MIBK) (Chiba and Kimura, 1989), tri-*n*-butyl phosphate (TBP), (Majumdar and De, 1960; Roddy et al., 1971), di(2-ethylhexyl) phosphoric acid (D2EHPA), (Sato et al., 1985; Roddy et al., 1971; Hirato et al., 1992; Kimura et al., 1984; Agatzini et al., 1986) and amines (Alguacil and Amer, 1986; Alguacil et al., 1987; Sahu and Das, 2000; Sahu and Das, 1997), are used for extracting iron (III). Metal recovery from liquid effluents has been studied by Andersson and Reinhardt (1983), Haines et al. (1973) and Tunley et al. (1976) mentioned the use of ion exchange resins to separate Fe(II) and Zn followed by Zn extraction by D2EHPA. Good and Bryan (1960, 1961) have extensively studied the extraction of some base metals including Fe(II) and Fe(III) in sulphate and chloride medium. Sanad et al. (1982, 1992) have studied iron extraction from chloride and bromide medium using tri-butyl phosphate. But these systems are found to have some disadvantages such as low extraction and difficult stripping, hence binary solvent mixtures have been tried. Though the literature on binary extraction of iron is very less, an attempt has been made to develop a suitable synergistic extraction process with mixed extractants that achieves improved iron(III) extraction from a concentrated iron solution in a sulphate medium.

2. Materials

2.1. Sample collection, solutions and reagents

10 L of sulphate waste pickle liquor was collected from Tata Tubes Ltd, Jamshedpur. The chemical analysis of this waste is given in Table 1.

The organic reagent used for the recovery of acid and iron in this study was Alamine 336, D2EHPA, MIBK. Alamine 336 (R_3N) was kindly supplied by Henkel and used as extractant without further purification. Alamine 336 is a mixture of saturated and straight chain trialkylamines with carbon chains C8 and C10, in which the proportion of the carbon chain C8 is about 2 to 1. It is a pale yellow liquid practically insoluble in water (<5 ppm) with an average molecular weight of 392 g/mol, a density of 0.81 g/cm^3, and a viscosity of 11 mPas (30 °C). Other solvents used as diluent and phase modifier are: distilled kerosene, MIBK, TBP, Benzene, Hexane.

Chemical analysis of the WPL, acid after extraction, iron and zinc in the solutions were done volumetrically by standard methods as mentioned in Vogel (1989). Mass balance gives the amount of acid extracted by the solvent. Trace metal analysis was done by atomic absorption spectro-photometer (Thermo SOLAAR S-2).

2.2. Experimental

Acid extraction from waste pickle liquor (WPL) of the steel tubes industry was done with 35% Alamine 336 (0.723 M) in kerosene. 10% of isodecanol was used as phase modifier. The loading capacity of the solvent for WPL containing 91.9 g/L was performed at 30 °C, 45 °C and 60 °C by multiple contact method at O/A ratio of 1:1. The organic and aqueous solutions were contacted for a period of 5 min to ensure complete extraction in a separatory funnel. The mixture then allowed to separate into organic and aqueous layers and the acid loaded organic was separated from the aqueous phase with residual acid. The same organic was shaken again with the fresh aqueous feed till the organic is completely loaded with the acid, which is indicated by the chemical analysis of the aqueous phase after each shaking. Various other parameters such as mixing time, O/A ratio, mixing

Table 1
Chemical analysis of the WPL of Tata Tubes Ltd, Jamshedpur

Constituents	H_2SO_4	Fe(T)	Fe(II)	Zn	Cu	Co	Ni	Mo	Mn	Cr
	g/L				ppm					
Composition	91.88	78.1	73.75	5.15	2.7	5.8	9.8	1	162	6.8

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