

# Recovery of Hydrochloric Acid from Spent Pickle Liquor by Evaporation

**Nikhil Yashwant .Ghare**

nikhil.ghare@rediffmail.com Gharda Institute of Technology, Lavel, Maharashtra State, India

\*Corresponding Author



<https://doi.org/10.55041/ijst.v1i1.001>

**Cite this Article:** .Ghare, N. Y. (2026). Recovery of Hydrochloric Acid from Spent Pickle Liquor by Evaporation. International Journal of Science, Strategic Management and Technology, *Volume 10*(01). <https://doi.org/10.55041/ijst.v2i2.135>

**License:**  This article is published under the Creative Commons Attribution 4.0 International License (CC BY 4.0), permitting use, distribution, and reproduction in any medium, provided the original author(s) and source are properly credited.

## Abstract:

Spent pickle liquor (SPL) of metal finishing industry is a major source of hazardous industrial wastes. Evaporation method has been investigated for the recovery of Hydrochloric acid from SPL. Recovery was studied with respect to time and efficiency.

Keywords: Spent pickle liquor, pickling, Evaporation , Hydrochloric acid

## 1.0 Introduction:

For efficient electroplating of zinc, pretreatment of steel surface is necessary; metal oxide and scales should be removed from the metal surface before electroplating. In order to clean the surface, the metal sheet, strip, wire etc. are pickled in hydrochloric acid or sulfuric acid or phosphoric acid bath. Carbon steels, with an alloy content less than or equal to 6%, are usually pickled in hydrochloric or sulfuric acid. Steels with an alloy content greater than 6% are pickled in two steps and other acids are used, such as phosphoric, nitric and hydrofluoric acid. For pickling of rust and acid-resistant chromium-nickel steels a bath of hydrochloric and nitric acid is used. Most copper alloys are pickled in dilute sulfuric acid, but brass is pickled in concentrated sulfuric and nitric acid mixed with sodium chloride and soot [1]. Steels are usually pickled in 20% HCl at 60–70°C. When the concentration of HCl becomes around 10% and the metallic impurities concentration such as Fe, Zn, Cr, etc. increases up to a certain level due to repetition of pickling in the same liquid, the pickle liquid becomes unusable and discharged as spent pickle liquor. Because of hazardous nature it cannot be disposed into land or water bodies without treatment. Conventional method of the pickle liquor disposal is acid neutralization with alkali, precipitation of metals and the sludge generated is disposed as landfill, or by evaporation and pyrohydrolysis of the pickle liquor. Due to this method there is acid loss as well as loss of metal values. Hence, it is necessary to develop a method to recover acid and iron values from these wastes[5].

Experiments were carried out to recover Hydrochloric Acid by evaporation and the results obtained are reported in this paper.

## 2.0 Materials and Methods

### 2.1 Materials

The spent pickle liquor (SPL) used in this study were containing Hydrochloric acid, it was procured from nearby electroplating industry. All the chemicals and reagents used in this study were of analytical grade and were of S D Fine Chemicals Ltd., Mumbai and Qualigens Chemicals, Mumbai.

### 2.2 Methods

#### 2.2.1 Physicochemical characteristics of SPL

Physicochemical analysis of SPL were determined using standard methods given in APHA (American Public Health Association) [2] and using the method given by F. Hasler and N. Stone [3,4]. This included free acid determination of HCl, determination of heavy metals like Fe, Zn, Pb, Cd, Ni and Cu, pH, Total Dissolved Solids (TDS), Total Suspended Solids (TSS), Specific gravity, Chlorides and Boiling point.

### 2.3 Recovery of acid(s) [5]

#### Analysis of Spent Pickle Liquor (SPL) containing Hydrochloric Acid:

Two samples were used in this case. The analysis of SPLs containing hydrochloric acid is depicted in Table 2.3.1 and Table 2.3.2. It was found that the SPL contains Fe in large quantity followed by Zn and Cu. The other metals include Pb, Cd, and Ni. The pH values recorded were 0.72 and 0.80 while free acid concentration was 20.1% and 9.2 % respectively in the two samples of SPL containing hydrochloric acid. It was also observed that concentration of Fe in samples was maximum than other metals followed by Zn, Cu, Pb, Ni, Cd respectively in decreasing order. Total dissolved solids (TDS), Total suspended solids (TSS) and chlorides values were 249 g/l, 0.1 mg/l, 248.1 g/l respectively and Boiling point recorded was 109°C for sample 1. Total dissolved solids (TDS), Total suspended solids (TSS) and chlorides values were 146.1 g/l, 0.1 mg/l and 145.72 g/l respectively and Boiling point recorded was 105°C for sample 2. The specific gravity was 1.07 and 1.05 for sample 1 and sample 2 respectively.

**Table 2.3.1: Analysis of SPL (Hydrochloric acid) from local steel industry (sample 1) (Evaporation Method)**

Component	Zn (mg/l)	Pb (mg/l)	Cd (mg/l)	Ni (mg/l)	pH	Fe (g/l)	Cu (mg/l)	Free acid (%)	TDS g/l	TSS mg/l	Chlorides g/l	Boiling Point °C
Concentration	153.58	14.56	0.82	6.49	0.72	40.67	46.25	20.10	249	0.1	248.10	109

**Table 2.3.2: Analysis of SPL (Hydrochloric acid) from local steel industry (sample 2)**

Component	Zn (mg/l)	Pb (mg/l)	Cd (mg/l)	Ni (mg/l)	pH	Fe (g/l)	Cu (mg/l)	Free acid (%)	TDS (g/l)	TSS (mg/l)	Chlorides (g/l)	Boiling Point 0C
Concentration	157.54	13.56	0.82	6.49	0.80	44.36	46.25	9.20	146.1	0.1	145.72	105

### Evaporation

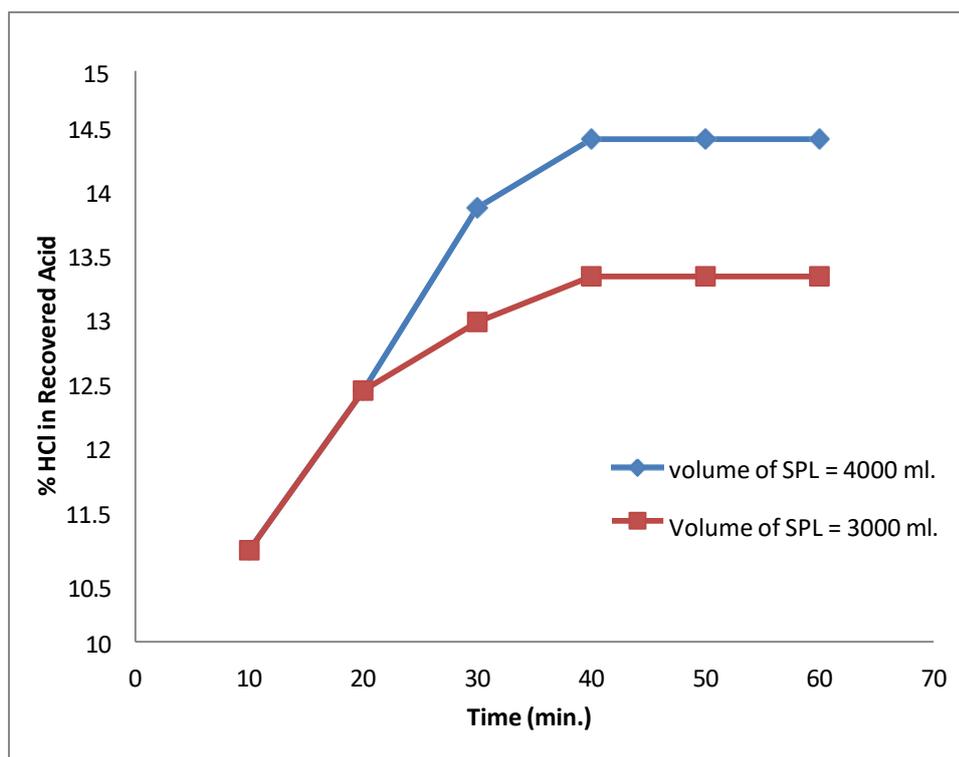
Evaporation process was carried out using evaporation apparatus which included 5 litre round bottom flask with heating mantle, a condenser & receiver [6]. A known volume of SPL containing hydrochloric acid sample was taken in the round bottom flask and the flask was heated till sample was boiled and formed vapours of acid. The vapours of acid formed were condensed and collected in a condensate receiver. The percentage free acid in condensate was estimated with time till constant reading was obtained.

**Table 2.3.3: Hydrochloric Acid recovery by evaporation (Volume of SPL = 4000ml)**

Sr. No.	Time (min.)	% Hydrochloric acid
1	10	10.80
2	20	12.20
3	30	13.80
4	40	14.40
5	50	14.40
6	60	14.40

**Table 2.3.4: Hydrochloric Acid recovery by evaporation (Volume of SPL = 3000ml)**

Sr. No.	Time (min.)	% Hydrochloric acid
1	10	10.80
2	20	12.20
3	30	12.80
4	40	13.20
5	50	13.20
6	60	13.20



**Fig. 2.3.1 Effect of time on concentration of recovered hydrochloric aci**

**Table 2.3.5: Analysis of Recovered Hydrochloric Acid by evaporation (Volume of SPL= 4000 ml.)**

Sr. No.	Parameter	Value
1	pH	0.60
2	Boiling Point	110 <sup>0</sup> C
3	Specific Gravity	1.10
4	Free Acid	13.20% w/v
5	Iron content	3.45% w/v

**Table 2.3.6: Analysis of Recovered Hydrochloric Acid by evaporation (Volume of SPL = 3000 ml.)**

Sr. No.	Parameter	Value
1	pH	0.90
2	Boiling Point	116 <sup>0</sup> C
3	Specific Gravity	1.17
4	Free Acid content	14.38% w/v
5	Iron content	2.75% w/v



**Fig 2.1: Evaporation Method**

**Result:**

The results shown in Table 2.3.3 and 2.3.4 for recovery of hydrochloric acid by evaporation indicate that concentration in recovered acid increased gradually with the time up to 40 min., however remained more or less constant thereafter (Fig. 2.3.1). The detailed analysis for the hydrochloric acid recovered by evaporation is presented in Tables 2.3.5 and 2.3.6. Hydrochloric acid recovery was found to be 90 percent.

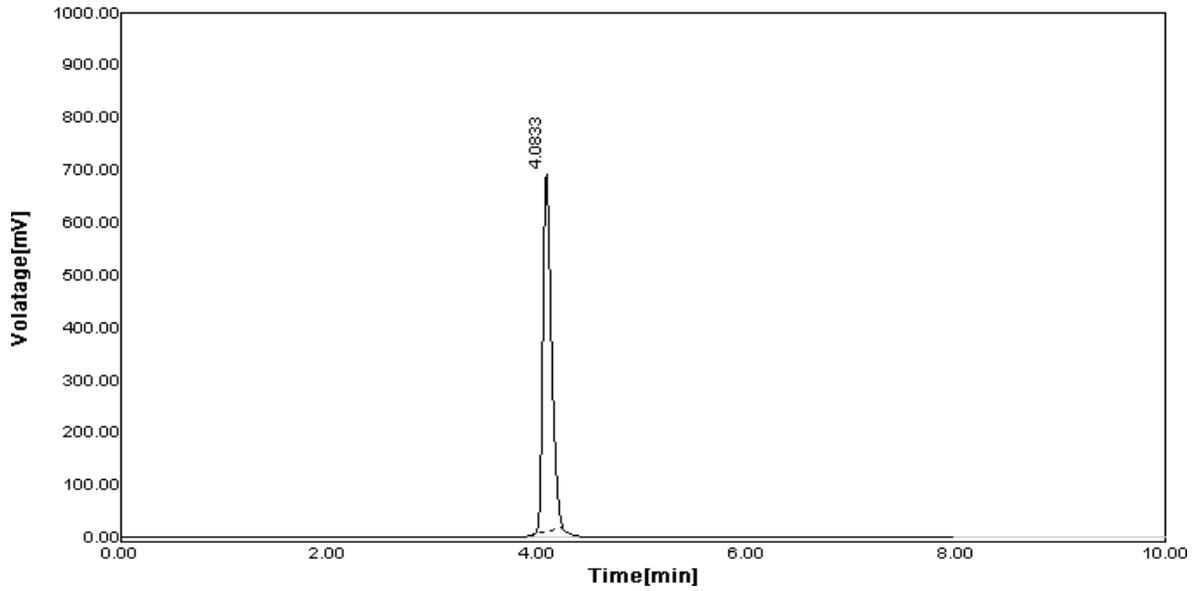
**2.4 Analysis of Recovered hydrochloric acid by HPLC method:**

The commercial hydrochloric acid used by the industry was analyzed qualitatively by HPLC method which was used as standard. The retention time was 4.083 min. and assay (purity) 100.00 %. It was within -5 % agreement. The recovered hydrochloric acid (2 samples) by evaporation was also analyzed qualitatively by HPLC method. Their

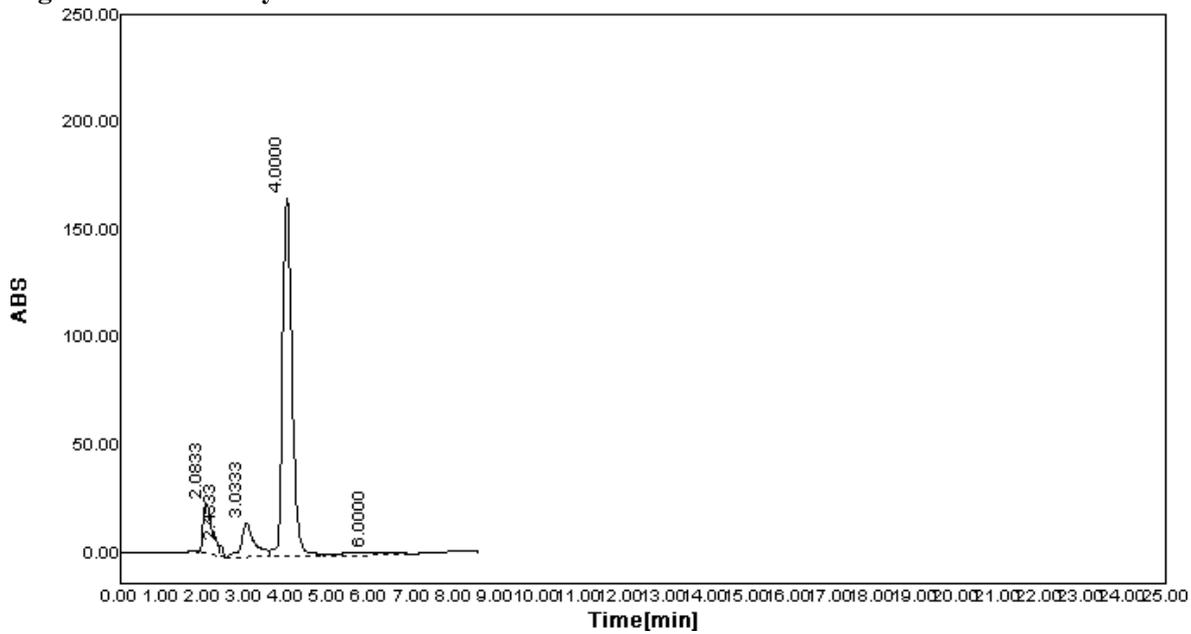
retention time was 4.00 min. and 4.48 min having assay (purity) as 70.78 % and 71.30 % as depicted in Table 2.3.7.

**Table 2.3.7: Analysis of Hydrochloric acid by HPLC method for Evaporation**

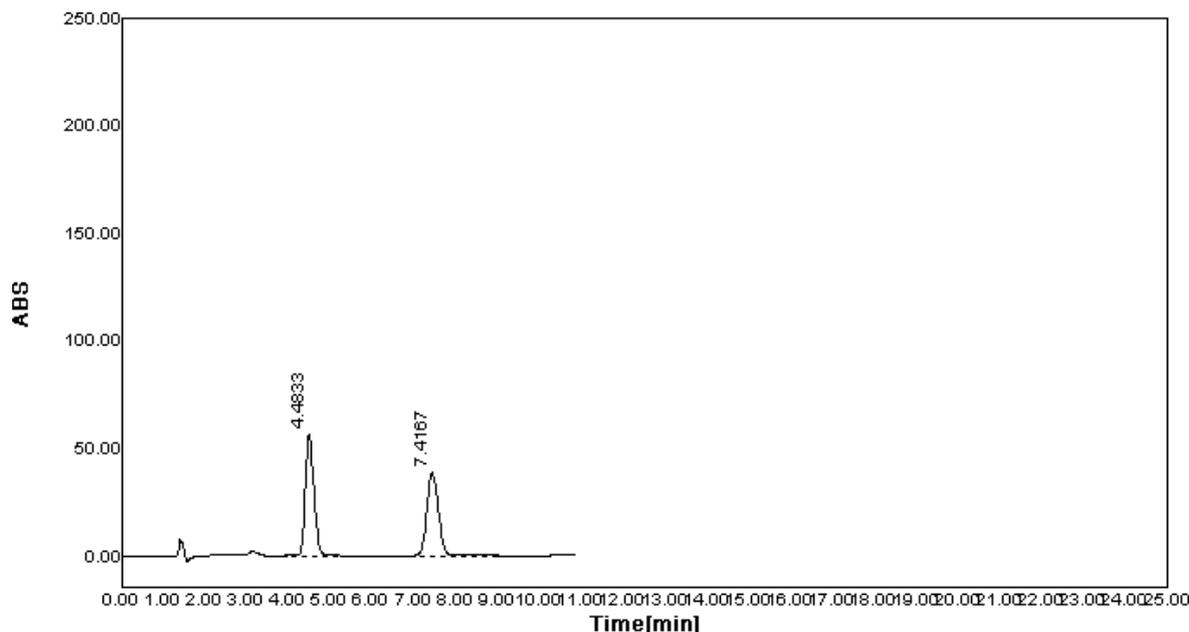
Sr. No.	Sample ID	Retention Time, RT, (min.)	% Purity
1	Standard	4.083	100.00
2	Sample 1	4.000	70.78
3	Sample 2	4.480	71.30



**Fig.2.2: Standard Hydrochloric Acid**



**Fig.2.3: Analysis of recovered hydrochloric acid (evaporation) by HPLC(Sample 1)**



**Fig.2.4 :Analysis of recovered hydrochloric acid (evaporation) by HPLC(Sample 2)**

The results obtained were in agreement with the reported values in the literature with some variations which may be due to the difference in concentrations of acids used for pickling, difference in the type of metal parts pickled, variation in climatic conditions etc. This analysis revealed that the spent pickle liquor (SPL) contained free acid and metals mainly Fe, Cu, Zn and Ni. The acid recovery for spent pickle acid by evaporation was medium. Also the method is energy intensive. The purity (% assay) of all recovered acid by evaporation was less.

#### **Acknowledgement:**

I am thankful to my family for inspiring me for this research work.

#### **References:**

- 1.Ghare N. Y., Patil V.S.and Wani K.S.. 2014.Recovery of Acids from Spent Pickle Liquor of a Steel Industry by Ion exchange Route *International Journal of Emerging Trends in Engineering and Development* , 4(1)
- 2.Clesceri L. S., Greenber A. R. and Eaton A. D. 1998. *Standard methods for the examination of water and wastewater,* 20<sup>th</sup> Edition, *American Public Health Association (APHA), American Water Works Association (AWWA) & Water Environment Federation (WEF)*, pp.3-12,3-13 and 2-24.
- 3.Hasler F. and Stone N. (1995), —The whys and hows of sulfuric acid pickling and recovery|, *Esco Engineering Ontario Booklet*
- 4.Hasler F., Stone N. (1997), *The Whys and Hows of Hydrochloric Acid Pickling*, ESCO Report
- 5.Ghare N Y, Wani K S, Patil V S .2013.A review on methods of recovery of acid(s) from spent pickle liquor of steel industry ;55(2):253-66.
- 6.Brazier K. Beecher 1999.Method of recovering hydrochloric acid from spent hydrochloric acid pickle waste , US4222997A.