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PERFORMANCES OF THE LOADS OF THE DUOLITE RESIN C 20 DURING THE DENICKELING OF COBALTIFEROUS SOLUTIONS (CASE OF SOLUTIONS OF HYDROMETALLURGICAL PLANTS OF SHITURU)

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Abstrat

The extraction of Nickel fromelectrolytic solutions of copper and cobalt treated at the hydromettalurgical plants of SHITURU, using DUOLITE resinC20, wascarried out according to two loadings of thisspecific nickel resin : the loading in bach and the loading in column.

The maximum bachloading of the nickel resinwascarried out at a pHof 2.5 for 6 hours, thusleading to a recoveryefficiency of nickel of about 98%. As for columnloading, the evolution of time shows that at a pH OF 2, for 8 hours, the recovery of is appreciable with a yieldwhich is close to 95%.

Abreviationslist

- pH : HydrogenPotential
- OF : Overflow
- UF : Underflow

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- PPS : Selectiveprecipitation
- ppm : part per million
- g/l : gram per liter
- mg/l : Miligram per liter
- GCM : General of quarries and mines.
- EW : Electrowinning
- DM : Middle start (Exhaustedelectrolyte solution)
- D2HPA : Di 2 ethylhexylpHospHoricacid
- CYANEX 272 : Di (2,4,4trimethylpentyl) thiopHospHinicacid
- P_{eff} : Weight of the metal in the effluent solution
- Pinf : Weight of the metal in the influent solution
- ND : Neutralization and Release
- S/L : Solid-liquid separation
- Mm : milimeter
- Ml : mililiter
- G : gram
- % : percentage
- US : SHITURU Plants
- H_2SO_4 : Sulfuricacid
- déFe : Deironing
- déCu : decoppering
- déCo : decobalting
- déNi : denickeling
- Nash : Sodium sulfhydrate
- NaOH : Sodium hydroxide
- rpm : Rotation Per Minute
- η_{Ch} : the recoveryefficiency at loading
- P_{inf} : the weight of the metal in the solution influences
- P_{eff} : the weight of the metal in the effluent solution

I. Introduction

Hydrometallurgical plants of SHITURU of the GCM in the province of HAUT-KATANGA in the Democratic Republic of the Congo have for main objective, the production of the copper by the electrolyticway. To this is added the production of Cobalt which is done by a bleedingperformed on the copper circuit.

The high purity of the cathodes produced is carried out afterseveral purification stepscarried out onthis line, namely : the deFe, the deCu, the deZn and the deNi. And thesedifferentconventional purification steps are done by the method of pps using lime milk to removeimpurities in the form of hydrates[1]. But giventhat the pHysicochemicalproperties of Co and Ni are similar, the Ni is stillfound in Cobbalt-rich solutions up to cobalt EW. In thisway, the nickel is not eliminatedsufficientlyduring the PPS, the Ni/Co ratio increases in the circuit with the risk of the position coding of nickel with cobalt.

To overcome the contamination of Co with Ni, a bleed is made on the Cobalt circuit where the Nickel is removed by precipitation in the form of Nickel sulphide by addingNasH. But the deNiyield is only about 50% and in addition, the use of NaSH causes the toxicity of the solutions.

In addition to the use of D2HPA and CYANEX 272 as Cobaltextractants, the deNi of the US Cobalt-rich solutions canalsobedone using a new reagent, DUOLITE C20.

[1] Roger RUMBU : Extractive Metallurgy of non-ferrousmetals. IndustrialPratices, p. 105 and p. 116, 2010

II. Problematic

The US GCM consists of several sections namely : the boom section, the leach section, the copperdecantation section, the copperelectrolysis tank house, the decobalting section and the cobalt electrolysis tank house.

Cobaltiferous solutions bleedingundergoesseveral purification stepsthatcanbedone by raisingpH, sulphidation or oxidation. And the recoveryefficiency of Cobalt in SHITURU plants by the PPS process has always been below 55%.

The operation of the elimination of the Ni by sulphurization is carried out using NaSHalsocausing Cobalt as shown by the followingreactions :

$NiSO_4 + 2\ NaSH \rightarrow NiS + Na_2SO_4 + H_2SO_4$

$CoSO_4 + 2\ NaSH \rightarrow CoS + Na_2SO_4 + H_2SO_4$

After the deNi using HaSH, the solutions resulting from the leaching after separation S/L on average titrate 12500 mg/l of Co and 150 mg/l of Ni. After deNi using NaSH, solutions are obtained which titrate 1000 mg/l Co and 80 mg/l Ni. This represents a deNi yield of about 50% with Cobalt loss of at least 20%[2].

In order to further improve deNi yield while minimizing Cobalt losses in nickel precipitates, even in terms of ppm, deNi of Cobalt-rich solutions should be carried out using the exchange purification method. The resin is a cationic exchange whose functional group is the sulfonate ion SO_3^{-}[3].

III. Characteristiccs of Cobalt solutions and reagents, equipment and procedures

The sample of the Cobalt-containing solutions of the US whichundergoes the deNi using the DUOLITE C20 resin is characterized by the chemical composition of Ni, Cu, Co, Zn and Fe, the concentration values of which are given in table 1 below :

Table 1 : Chemical analysis of Cobalt-rich US solutions at pH = 4.2

Element	**Ni**	**Co**	**Cu**	**Zn**	**Fe**
Concentration [g/l]	0,16	11,6	0,01	0,08	0,01

Reagentsused to purify Cobalt electrolyte solutions are :

[2] Roger RUMBU : Extractive Metallurgy of non-ferrousmetals. IndustrialPratices, p. 105 and p. 120, 2010

[3] CK GUPTA : Extractive Metallurgy of Molybdenum, p. 190, 1990

- 0.1 N NaOH solution (normal) to fix the pH ;
- A 98% sulfuricacid solution to prepareacidulated water for use in conditioning the resin ;
- Distilled water for the dilution of sulfuricacid and the preparation of the solution of caustic soda ;
- The DUOLITE resin C20 used for the nickel removal of Cobalt-rich solutions is presented in table 2 below :

Table 2 :

Functional group	**Sulfonate SO_3^-**
Particle size D_{95}	35 mesh (0,42 mm)
Color	white
Apparent dry density	0,73 g/l
Apparent density in water	1,012 g/l
Regeneration time with sulfuricacid at 50 g/l	2 hours
Rinsing time with distilled water	1 hour
$\frac{\textbf{acid solution volume}}{\textbf{resin solution volume}}$	2

Characteristics of DUOLITE resin C20

The main reaction of ion extraction by resin is given by : $M^{m+} + RnH^{+} \rightarrow MR^{m+} + nH^{+}$ where M is the metal to beextracted, and R is the functional group of the resin[4].

The equipmentused in the deNi tests consists of instruments, glassware and certain accessories.

Instruments :

- A mechanicalagitator brand IKA Labotecchnik (RW 20DZ)
- The seriessives, type TYLER, of mesh : 10, 20, 28, 35, 65 and 100 corresponding in micrometer, respectively to 1500, 750, 535, 429, 231 and 150.
- A METROHM 713 PHmeter

[4]Ray E BOLZ Technology & Engineering pg 187 :1973

- A stopwatch

Glassware :

- 600 ml beakers, burettes, graduated test tubes of 100 ml and 1000 ml.
- 2000 ml flat-bottomed ballons

Otheraccessories :

- A pipette, threefunnels, flasks, flaps, a stand, filterpaper
- A column
- Serie of sieves, type TYLER

The procedures for deNi cobalt solutions are summarized in two types of test : the DUOLITE resin C20 conditioning test and the Bach and columnloading tests of the Ni and Co resin.

The main objective of the resinconditioning test was to bereassured y the reactivity of the DUOLITE resin C20, whichconsists in correctly fixing the main impuritywhich is nickel. This conditioningconsisted first of all in makinggranulometricanalysiswhichaim at carrying out a narrower calibration of the grams of the resin and theneluting with sulfuricacidfollowed by rinsing.

Afterrinsing, the resinwasregenerated with a solution of sulfuricacid at 50 g/l to ensureits initial state. To remove traces of residualacid, DUOLITE resin C20 wascontacted with distilled water.

The tests for resinbachloadingwerecarried out as follows ;

- Adjust the pH of the solution to bepurified to the set point ;
- Pour the resinintoabeaker ;
- Add in the beaker 200 ml of the cobalt solution to bepurified ;
- Place the stirrer and activateit ;
- Stop stirringafter 60 minutes ;
- Separate the twoseparatedpHases by filtration ;
- Takeasample of the raffinate by chemical analysis ;
- Resume the test for oother pH values.

The loading tests of the columnresinwerecarried out as follows :

- Pour 375 mg of DUOLITE C 20 into a 750 ml column ;
- Adjust the pH of the cobalt solution to bepurified to the desired value.
- Allow the solution fo flow through the resinbed ;

- Stop the operation for a definite time ;
- Takeasampleraffinate by chemical analysis ;
- Repeat the test for otherpH values

IV. Presentation and discussion of the results

The conditioning of the resinbegan with particle size analysis, the results of which are presented in the table below :

Table 3. Granulomericanalysis of DUOLITE resin C20

Sieve size		**Refusal Mass [g]**	**Refusalpercentage [%]**	**Cumulative refusalpercentage [g]**
Mesh	**Mm**			
10	1500	0	0	0
20	750	88,95	22,65	22 ;65
28	535	214,48	54,61	77,26
35	429	75,24	19,16	94,62
65	231	14,02	3,55	99,97
100	150	0,04	0,01	99,98
< **100**	0,429	0,005	0,0013	100
Total			100	

We notice that about 95% of particles of DUOLITE C20 are found to belargerthan 35 mesh.

Afterrinsing and regenerating the resin with acid solution at 50 g/l for 2 hours, the loading tests in bach with PH variation, werecarried out under the following conditions :

- Contact time : 60 minutes ;
- Temperature : 25° C ;
- Stirring speed of the cobalt solution : 800 rpm ;
- Initial concentration of Ni and Co in the stock solution : 1600 mg/l and 11 620 mg/l ;

- Volume of the resin : 100 ml corresponding to 50 g ;
- PH variation in the followingorder : 0,5 ; 1 ; 1,5 ;2 ; 2,5 ;3 ; 3,5 ;4 ;4,5 ;5.

The performances of the extraction of the DUOLITE resin C 20 at the differentPH values weredetermined by the calculations :

- Of the recoveryyield at loading of the resingiven by the ratio = $\eta_{Ch} = \frac{P_{inf} - P_{eff}}{P_{inf}} 100$
- of the Co/Ni ratio in the effluent solution :$\frac{cobalt\ weight}{Nickel\ weight}$ ou $\frac{Co}{Ni}$

the values of thesetwoquantities as well as theirgrapHs for differentpH values are presented in table 4 and Figure 1 below :

Table 4 : Values of the recoveryyields of Ni and Co as well as those of the ratio at the loading in bach of the resin as a function of the PH

pH	**Residual concentra »tion[mg/l]**		**Chargedmetalweight[mg]**		**Recoveryefficiency[%]**		**Ratio $\frac{Co}{Ni}$**
	Ni	**Co**	**Ni**	**Co**	**Ni**	**Co**	
0,5	106,4	10117,4	10,7	296,50	33,5	12,8	95,1
1	29 ;8	8173,36	26,03	685,3	81,3	29,5	273,8
1,5	9,9	7236,1	30,2	872,8	94,3	37,6	787,9
2	3,3	6543,6	31,34	1011,29	97,9	43,6	1994,9
2,5	3,1	5949,64	31,4	1130,1	98,1	48,7	1916,8
3	2 ,7	3679,5	31,4	1584,1	98,2	68,3	1321,7
3,5	2,9	2959,2	31,4	1728,2	98,2	74,5	1010,6
4	2,9	2932,9	31,4	1735,6	98, 2	74,5	1014,6
4,5	2,7	2932,9	31,4	1735,8	98,2	74,8	1019,8
5	2, 8	2945,5	31 ;4	1750,9	98,2	75,5	999,1

Figure 1 : Evolution of Ni and Co recoveryefficiencies at bachloading as a function of pH

The analysis in Table 4 and the grapH in Figure 1 shows thatduring the DUOLITE resin C20 loading, Ni recovery is higherthanthat of Co. This canbeseen in the appreciable value of 98 % of the recoveryyieldfrom the PH OF 2,5 whilethat of the Co is only 48,5%.

Beyond PH 2,5 recovery of Ni becomes constant, but that of Co continues to increase. In addition, it is when the Co/Ni ratio in the effluent becomes high that Ni recoveryreachesits high value. It is thereforeappropriate to retain the value of 2,5 as the optimum PH to maintainwhenloadingbach DUOLITE resin C20 Nickel.

The conditions of the columnloading tests are as follows :

- columnloading time : 8 hours
- temperature : 25° C
- flow direction of Cobalt solution : form top to bottom
- initial concentration of Ni and Co in stock solution : 1600 mg/l and 11620 mg/l
- volume of the resin : 750 ml corresponding to 50 g pH of the solutions with deNi : 0,5 ; 1 ; 1,5 ; 2 ; 2,5.

The results of columnloading tests werepresented in Tables 5, 6, 7, 8, 9, 10 and Figures 2, 3, 4, 5, 6, 7, 8, below

Table 5. Values recoveryyields of Ni and Co as well as those of the ratio $\frac{Co}{Ni}$ at PH = 0,5

Time [hour]	**Volume of influent corresponding effluent [m/l]**	**Residualconcentrtion [mg/l]**		**Weight of the metal in the effluent[mg]**		**Weight of the metal in the effluent [mg]**		**Recoveryefficiency [%]**		**Ratio $\frac{Co}{Ni}$**
		Ni	**Co**	**Ni**	**Co**	**Ni**	**Co**	**Ni**	**Co**	
1	800	120	10210	96	8168	128	9280	25	11,9	85,1
2	1550	126,5	10327	196	16010	248	17980	20,9	10,9	81, 7
3	2300	131,0	10438	301	24010	368	26680	18,2	10	79,8
4	3010	132,7	10492	400	31586	482	34920	17	9,6	78,7
5	3800	133,7	10542	508	40067	608	44080	16,4	9,1	78,9
6	4520	134,5	10587	608	47852	722	52430	15,9	8,7	78,7
7	5280	134,1	10624	713	56093	845	61250	15,6	8,4	78,7
8	6060	135,6	10675	822	64690	970	70296	15,2	7,9	78,7

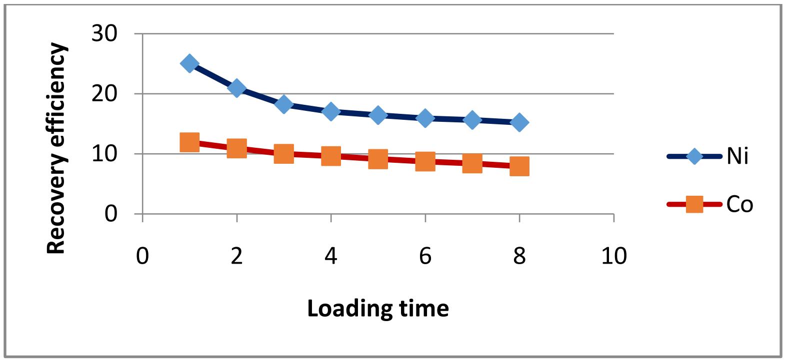

Figure 1 : Evolution of Ni and Co recoveryefficiencies at columnloading as afunction ofPH = 0,5

Table 6. Values of the Ni and Co recoveryyields as well as those of the ratio at pH = 1

Time [hour]	Volume of influent corresponding to effluent [m/l]	Residual concentration [mg/l]		Weight of the metal in the effluent [mg]		Weight of the metal in the influent [mg]		Recoveryefficiency [%]		Ratio $\frac{Co}{Ni}$
		Ni	Co	Ni	Co	Ni	Co	Ni	Co	
1	800	9	5580	7,2	4464,0	128	9280	94,4	52,0	620
2	1559	9,3	6876,7	14,5	10658,9	249	18084	94,3	49,0	735
3	2305	9,5	7777,8	21,9	17889,0	369	26738	94,1	33,4	817
4	3009	11,6	8128,6	34,9	24548,3	481	34904	92,7	29,8	703
5	3410	12,7	8360,8	43,3	31770,9	546	39556	92,1	19,7	734
6	4529	13,6	8533,3	61,6	38570,6	725	52536	91,5	26,6	626
7	5524	14,5	7767,9	80,1	43577,8	884	64978	90,9	31,0	544
8	6290	16,1	7026,1	100,7	44193,9	1006	72964	89,9	39,4	439

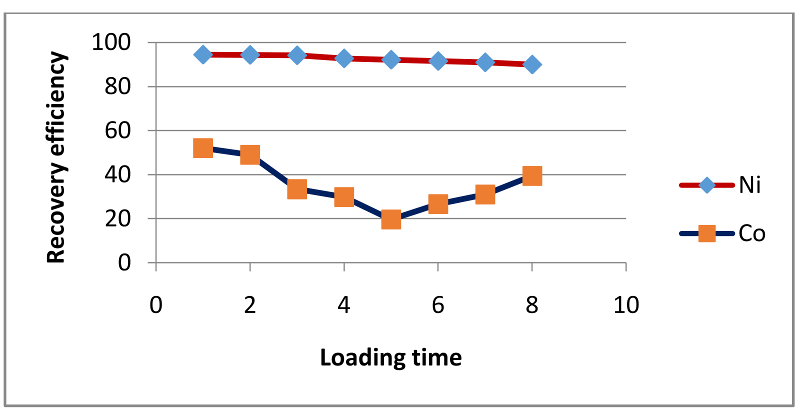

Figure 2 : Evolution of Ni and Co recoveryefficiencies at columnloading as afunction ofpH = 1

Tableau 7 : Vaalues of the Ni and Co recoveryyields as well as those of the ratio $\frac{Co}{Ni}$at pH = 1,5

Time [hour]	Volume of influent corresponding to effluent [m/l]	Residual concentration [mg/l]		Weight of the metal in the effluent [mg]		Weight of the metal in the influent [mg]		Recoveryefficiency [%]		Ratio $\frac{Co}{Ni}$
		Ni	Co	Ni	Co	Ni	Co	Ni	Co	
1	800	5,8	5510	4,6	4408	128	9280	96,6	52,5	950
2	1559	5,9	6212	9,2	9628,6	249	18084	96,3	46,8	1047
3	2300	6,0	6734,7	13,8	15489,7	368	26680	96,3	41,9	1122
4	3010	6,6	7109,3	19,9	21469,9	483	35032	95,9	38,7	1079
5	3800	7,5	73366,9	28,5	27994,5	608	44080	95,3	36,5	982
6	4520	8,4	7587,9	37,9	34297,3	723	52432	94,8	34,6	905
7	5280	9,1	7749,6	48,1	40917,7	846	61306	94,4	33,3	852
8	6060	9,9	7920,7	59,9	47999,5	970	70296	93,8	31,7	800

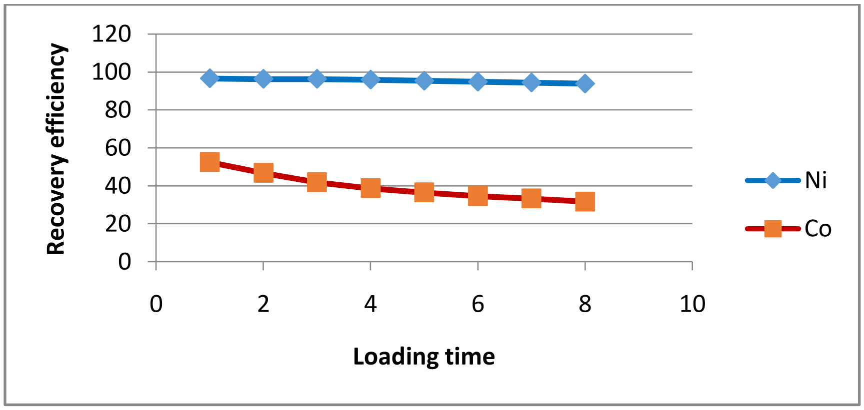

Figure 3 : Evolution of Ni and Co recoveryefficiencies at columnloading as a function ofpH = 1,5

Table 8 : Values of the Ni and Co recoveryyields as well as those of the ratio at pH = 2

Time [hour]	Volume of influent corresponding to effluent [m/l]	Residual concentration [mg/l]		Weight of the metal in the effluent [mg]		Weight of the metal in the influent [mg]		Recoveryefficiency [%]		Ratio $\frac{Co}{Ni}$
		Ni	Co	Ni	Co	Ni	Co	Ni	Co	
1	800	8,2	5440,0	6,6	4352,0	128	9280	94,8	53,1	659
2	1553	8,5	5711,5	13,2	8852,8	249	18015	94,7	50,9	671

3	2333	8,7	6324,7	20,3	14673,3	373	27063	94,6	45,8	723
4	3045	8,9	6696,1	27,1	20356,1	487	35322	94,4	42,4	751
5	3843	8,9	6948,4	34,2	26681,7	615	44579	94,4	40,1	780
6	4607	8,9	7258,2	41,0	33097,6	737	53441	94,4	38,1	807
7	5320	9,1	7492,5	48,4	39860,4	851	61712	94,3	35,4	624
8	6100	9,2	7727,0	56,1	47134,8	976	70760	94,4	33,4	840

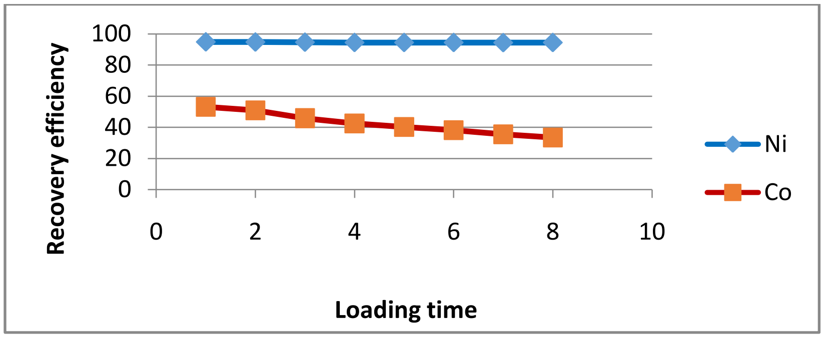

Figure 4 : Evolution of Ni and Co recoveryefficiencies at columnloading as a function of pH = 2

Table 9 : Values of the recoveryefficiencies of Ni and Co as well as those of the ratio $\frac{Co}{Ni}$ at PH = 2,5

Time [hour]	**Volume of influent corresponding to effluent [m/l]**	**Residual concentration [mg/l]**		**Weight of the metal in the effluent [mg]**		**Weight of the metal in the influent [mg]**		**Recoveryefficiency [%]**		**Ratio** $\frac{Co}{Ni}$
		Ni	**Co**	**Ni**	**Co**	**Ni**	**Co**	**Ni**	**Co**	
1	800	8,1	5500	6,5	4400	128	9280	94,0	52,6	679
2	1551	8,5	5748,5	13,2	8910,2	248	17992	94,7	50,5	676
3	2320	9,1	5928,7	21,1	13754,6	371	26912	94,3	48,9	652
4	3040	9,7	6369,3	29,5	19359,6	486	35264	93,9	45,1	656
5	3840	1,02	6680,1	39,2	25641,6	614	44544	93,6	42,4	654
6	4560	10,8	7031,5	49,3	32063,9	730	52896	93,2	39,4	651
7	5320	11,2	7212,9	59,6	38373,1	851	61712	93,0	37,8	644
8	6100	11,7	74445,8	71,4	43415,6	976	70760	92,7	35,8	636

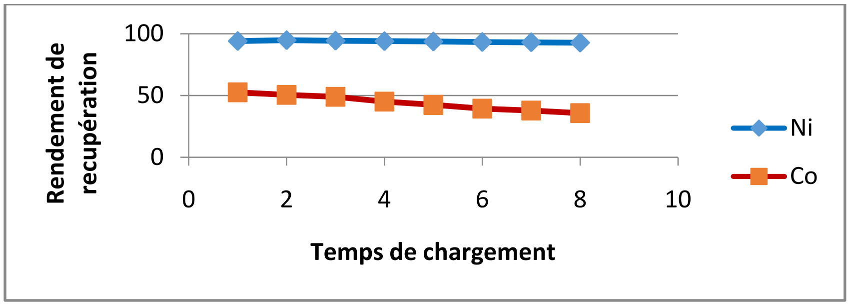

Figure 5 : Evolution of Ni and Co recoveryefficiencies at columnloading as a function of pH = 2 ,5

In view of theseresults, the columnloading of the Ni resin is carried out with recoveryefficiecies of around 90%. AlreadyfromPHquals 1 but the ratio in the effluent solution is stilllow, ie 439. But for a Cobalt solution at PH 1,5 ; the recoveryefficiency in Ni reaches a maximum of 96% with a high ratio of 1122 after 3 hours in the effluent solution. This is veryinterestingbecause at the end of this test the recoverydid not varymuch ; itremainedalmost constant.

Tests carried out at PH = 2 show that the residual concentration of Ni aftereachhour and stilllow and the efficiency of deNi is almost constant of the order of 94%. And after 8 hours, weobtainasignificant volume of effluent with a good ratio of 840 which is a high value compared to thatobtainedafter the same test duration at PH = 1,5. And the Cobalt recoveryefficiencyreamainslow, ie 33 ,4%

V. Conclusion

The nickel removal of the cobalt-rich solutions from the hydrometallurgical plants of SHITURU using DUOLITE resin C20 is easilycarried out during the bachloading with a Ni recoveryefficiecy of about 98% with a cobalt-rich solution at a PH OF 2. Titrating 160 mg/l of nickel. With the same solution, the nickel removalperformedduringcolumnloading at aPH of 2 gave a recoveryefficiency of 95% and an appreciable ration of 840 after 8 hours.

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