Recycling Possibilities of Spent Potlining Carbon Material Generated from NALCO's Smelter

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Abstract

Total 6 000–7 000 tonnes of Spent Potlining Material (SPL) is generated from aluminum electrolysis process of NALCO's smelter each year, out of which around 45–50 % is carbon portion (first cut) and rest is refractory portion along with other insulating lining materials. SPL has been classified as hazardous waste by Indian environmental regulations due to the presence of mainly leachable fluoride and cyanide constituents. Safe disposal of this unavoidable but hazardous waste product is a challenge faced by NALCO. This paper focuses on laboratory scale investigation on SPL carbon portion (first cut) for finding its recycling possibilities after treatment. The major constituents of SPL carbon portion (first cut) are carbon, fluorides, sodium compounds, along with minimum amount of sodium cyanides and other impurities. The basic process involves pressure leaching of ground SPL in water to remove cyanides, recovery of sodium as sodium hydroxide solution, recovery of fluorides as calcium fluoride and possible utilization of treated carbon in prebaked anode matrix.

Key words: Spent pot lining, Pressure leaching, Recovery of fluorides and carbon, Prebaked anode.

1. Introduction

In the Hall-Héroult smelting process, aluminum oxide is dissolved at 960 °C in a molten electrolyte composed of cryolite, aluminium fluoride, calcium fluoride, and some other minor compounds; dissolved alumina is electrolyzed to give aluminum metal at the cathode and oxygen gas at the anode [1].

In the aluminium production pot, the electrolyte and the molten aluminium are contained in a steel shell lined with refractory and thermally insulating material at the bottom and carbon cathodes and side slabs sealed with ramming paste at the upper layer of pot lining. Graphitic, graphitized or semi-graphitized materials are now used extensively as prebaked carbon cathode blocks. The other materials used are semi-graphite and silicon carbide (SiC) sidewall bricks and carbonaceous ramming paste. Steel collector bars are embedded in the carbon cathode to conduct the electric current away from the pot. Figure 1 shows a schematic drawing of an aluminium electrolysis pot.

Insulation bricks and refractory bricks are used to insulate the cathode thermally. These bricks are porous and vulnerable to penetration of electrolyte components through the cathode blocks. The real acting cathode from an electrochemical point of view is the top surface of the molten aluminum pool.

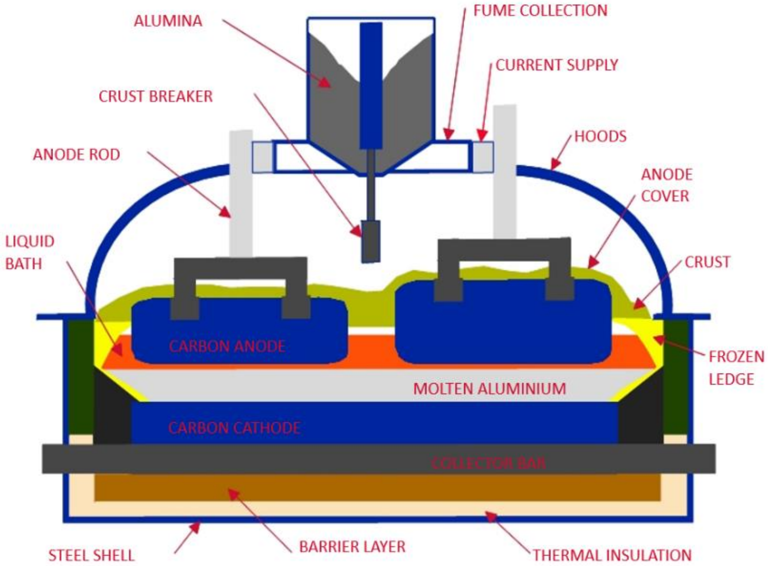

Figure 1. Schematic drawing of an aluminum electrolysis pot.

Pots are now typically 9–18 m long, 3–5 m wide, and 1–1.5 m deep. The depth of the operating pot cavity is relatively low, about 0.4–0.6 m. Carbon is the material chosen to withstand best the combined corrosive action of molten fluorides and molten aluminium. However, in actual operation, the frozen electrolyte protects the sides of the pot. Nowadays silicon carbide is used as sidewall material, but this material is also corroded by the electrolyte and needs to be protected.

The pot is shut down when the damage to the cathode does not allow it to continue its operation, usually after a period of 5–10 years. The material generated after removing bath material and aluminum metal and separating cathode bars is called spent pot lining, comprising carbon materials (first cut) originating from the cathode blocks, side blocks and lining paste and the refractory and insulating materials (second cut).

The spent lining material, which is composed of carbon, refractory material and cryolite, including fluorine, aluminum, sodium, calcium and silicon values, along with free and complexed cyanides, carbides and nitrides, is hazardous due to leachable fluorides and cyanides and must be treated safely. The safe disposal of spent linings has for a long time presented a challenge to the industry. That challenge continues with ever stricter environmental standards. Thus, disposal residues are limited to very low concentrations of fluorides and cyanides.

There is no global sustainable solution for management of SPL. The disposal and treatment practices adopted for SPL is highly diversified, ranging from sea deposits, secured landfill to dedicated plant and region specific valorization plants. SPL treatment costs vary widely and they mainly depend on the type of process and product use or reuse and regional regulations.

As per reports, few aluminum companies in India and abroad have started recycling SPL in cement industries and steel industries, third party alternatives exist to treat SPL [2].

There are two general categories of technology, i.e., technology that treats and stabilizes the waste (rendering the waste harmless) and enables landfilling of the stabilized-waste, and technology that focuses on making products out of the waste. There are hydrometallurgical and pyro metallurgical options for both categories.

NALCO has been taking utmost care for safe storage and disposal of SPL like many other aluminium smelters. This paper outlines the storage and disposal practices followed by NALCO along with research efforts for finding a suitable process for treatment and reuse of SPL

One such research and development (R&D) work is described in this paper which involves treatment of SPL carbon portion generated in NALCO's smelter plant by hydrometallurgical route for removal of CN and recovery of fluoride and sodium values and recycling possibilities of carbon residue generated from the process.

2. Spent Potlining and Its Formation

During the operation of the pot, the cathode is being continuously penetrated by sodium and bath components. Sodium generated from the sodium fluoride and elemental aluminium reacts with carbon forming intercalation compounds and attacks the lining materials. Impregnation of sodium causes a large increase in volume of the whole solid system. The bath can react or crystalize without reacting. In the latter case a very strong crystallization pressure appears. Under this condition, the cathodic system is gradually disrupted due to the swelling and heaving of the solid mass. Due to this reason, the pots are to be closed for re-lining after operating for a period of time which can vary from five years to as long as ten years. Carbon potlining deforms to such an extent that normal operation becomes impossible and consequently the pot has to be shut down to replace the whole lining. The potlining residue dug out from the pots, before the latter are relined, is commonly called "spent potlining" (SPL). The upper layer - the carbonaceous part - consisting of cathodes, side slabs and ramming mass is called first cut spent potlining and the bottom layer comprising of mainly refractory and insulating bricks along with alumina and other insulating materials is called second cut of SPL. Older pot technologies typically produce 20-30 kg SPL per tonne of aluminium whereas modern high amperage pot technologies typically generate 16-20 kg SPL per tonne. SPL generated is heterogeneous and its composition varies with type of technology, age of pots, operational practice, etc.

2.1. Chemical Reactions within the Potlining

Details of expected reactions [3], which cause the deformation of cathode lining material during the course of metal production in the pot, are discussed in this section.

Oxidation of carbon in air as expressed by the following Equation (1), causes the deterioration of the lining:

$$C(s) + O_2(g) = CO_2(g) \tag{1}$$

Air oxidation is enhanced by the presence of impurities such as salts of sodium and by high porosity (in the pore size range of 0.15–15 µm) and inhibited by halogens like fluorine. Air oxidation is also accelerated by increase in temperature, making thermal conductivity and insulation design important parameters. The side lining may be oxidized from either the bath side, the top or from the back side. The liquid aluminium pool will usually protect the bottom against oxidation.

The reaction between aluminium metal and carbon leading to the formation of aluminium carbide is spontaneous at temperatures of ~ 970 °C as expressed below at equation (2):

$$4Al(1) + 3C(s) = Al_4C_3(s) \quad \Delta G^0 = -147 \text{kJ/mole at } 970\ °C \tag{2}$$

The high negative value of the standard Gibbs free energy change, ΔG^0, indicates that the above reaction is thermodynamically favored in the forward direction at all temperatures of concern in electrolytic aluminium production. During electrolysis, the dissolved carbide in the melt may be oxidized by the anode gas as shown at Equation (3)

$$Al_4C_3(sol) + 9CO_2(g) = 2Al_2O_3(sol) + 12CO(g) \tag{3}$$

This leads to dissolution of the carbide films. The subsequent under saturation given in equation (4) may lead to a steady consumption of cathode carbon.

$$6NaF\ (l, sol) + 34Al_2O_3(sol) = 3Na_2O.11Al_2O_3\ (s) + 2AlF_3(l, sol) \tag{4}$$

If air is allowed to enter the carbon lining the following four reactions (5-8) may occur

$$4Na3AlF6\ (l) + 12\ Na(in\ C) + 3O_2(g) = 2Al_2O_3\ (S) + 24\ NaF\ (l) \tag{5}$$
$$4Na\ (in\ C) + 3O_2(g) + 2C(s) = 2Na_2CO_3(s,l) \tag{6}$$
$$2Na(in\ C) + 2C(s) + N_2(g) = 2NaCN(l) \tag{7}$$
$$2Al(l) + N_2(g) = 2AlN(s) \tag{8}$$

The concentration of NaCN is usually more in end and side linings of the pot which may contain over 90 % of all cyanides in SPL.

3. SPL Generation and Disposal Practice in NALCO Smelter Plant

The AP18 technology pots in NALCO smelter plant used for aluminium electrolysis are made of steel shells lined with refractory bricks, alumina and other insulating materials at the bottom layer and carbon cathodes sealed with tepid ramming paste and carbon side slabs at the upper layer of the lining.

With an installed capacity of 960 pots, the generation of SPL is around 5000–6000 tonnes per year with 2500–3000 tonnes first cut and the rest is second cut. SPL has been classified as a hazardous waste mainly due to presence of leachable fluorides and cyanides. The composition of SPL is also variable depending on the age of pots, type of cathodes, type of other lining materials used and concentration of specific impurities in different pockets of bottom lining.

SPL is removed from NALCO pots by wet digging methods. After removing the crushed bath and alumina, aluminium metal pad from the dead pot, water is poured into the pot cavity. The carbon material is excavated after 24 hours of soaking. During water soaking period (for 24 hours), shunted pots are covered with a big single pot shell hood, for arresting release of gases in to working site. The single potshell hood is connected with a "sprinkler system” for suction of gases and released to atmosphere through a chimney. SPL material is transported to designated sheds for storage and further disposal. Figure 2 shows the pictures of a newly lined pot and dead pot lining.

NALCO’s smelter started its operation during 1987 and the SPL material generated from the plant’s first-generation pots were stored in secured cemented lined capsules under the ground after treatment with stabilizing chemicals. Since the last decade the SPL material is stored under covered sheds as per the government guidelines which restricts the landfilling of carbon portion of SPL due to its high calorific values. Over the years some SPL have been disposed to authorized re processors and external agencies for disposal.

Figure 2. Left: newly lined pot lining, Right: dead pot lining.

4. Research and Development Efforts on NALCO's SPL

Several experimental research works have been carried out by NALCO to find out a sustainable process for treatment and reuse of SPL. Studies carried out and findings and limitations are tabulated in Table 1.

Table 1. Previous works carried out at NALCO.

Name of the study carried out at NALCO	Findings and limitations
Using carbonaceous portion of spent pot lining as a co-fuel.	Minimal quantity 1–2 % SPL carbon can be blended in coal for co-firing in boilers. However the grindability of SPL and presence of F and Na was a concern.
Utilization of refractory portion of spent potlining for manufacturing of cement.	This study concluded that 1–2 % SPL refractory can be added during clinker production which helps in lowering the clinkerisation temperature and F acts as a mineralizer.
Possibility of producing carbon electrode paste from SPL	20–30 % SPL carbon after water leaching may be possibly utilized during making of tepid ramming paste.
Decontamination and recovery of carbon value from spent pot lining material – A pilot plant study.	Scaling up found difficult due to use of corrosive per chloric acid and cost of treatment was high.
Study of heat treatment process for destruction of toxic cyanide present in NALCO`s SPL	The study was limited to only removal of leachable cyanide from SPL carbon. A commercial plant based on the technology has been set up for detoxification of SPL carbon.

Based on the findings of past R&D work and literature studies [4–7], attempts have been made to find a cost effective and viable option for treatment of SPL carbon to extract all valuable constituents and utilize them gainfully. In the present work efforts have been made to treat the SPL carbon with water and recover NaOH at a particular concentration after removal of fluoride by lime treatment. Reuse of carbon residue in anode making and NaOH in alumina refinery plant has been explored.

As known green anodes are made from approx. 85 % of CP coke and recycled anode butts combined and around 15 % pitch. In NALCO smelter around 300 000 tonnes of green anodes are produced per year. Total quantity of SPL carbon portion being generated is around 2500 tonnes. Experiments were carried out to know the quantity of SPL carbon that can be blended in anode recipe without affecting its quality.

4.1 Phase-1 Experiments

A representative samples of spent potline carbon portion was ground and characterized for the following chemical composition as given in Table 2.

Table 2. Chemical composition of SPL.

Component		Quantity (%)
Carbon		50-55
Fe_2O_3		0.5-1
Na_2O		13-15
SiO_2		0.2-0.7
CaO		0.2-0.35
F		10-15
CN		0.10-0.12

Table 3. Major phases identified in SPL.

Component	Phase
Fluorides	Na_3AlF_6, NaF, CaF_2
Cyanides	NaCN, $NaFe(CN)_6$
Aluminium	Al_2O_3, $NaAl_{11}O_{17}$
Carbon	Graphite
Sodium	Na_3AlF_6, NaF
Calcium	CaF_2
Iron	Fe_2O_3
Si	SiO_2

Bench scale experiments were carried out by making bench scale anodes [8] from batches of 5.5 kg of carbon anode materials comprising of calcined petroleum coke, anode butts and coal tar pitch. Recipe comprised of 20 % very coarse particles fraction (4.75 mm–13.2 mm) , 22 % coarse coke (1.7 mm–4.75 mm), 20 % medium coke (0–1.7 mm), 9 % other recycled butts (0–8 mm), 29 % fines (–200 mesh > 70 %) , 14.5 % coal tar pitch.

Seven batches of the recipe were prepared with varied addition of ground SPL (composition in Table 2) starting from no addition up to 4 % addition in other recycled butts fraction as shown in Table 4, for making bench scale anodes. Bench scale anodes were prepared by mixing the recipe at 173-175 °C and pressing to get 50 mm diameter and 110 mm length (330–340 g) anodes for each of the seven batches. The anodes were baked at 1100 °C in bench scale anode baking furnace. The properties of green anodes and baked anodes were tested and given in Table 4.

Observations: Influence of SPL carbon additions on anode density is shown in Figure 3, on reactivity behavior is shown in Figure 4 and on impurities is shown in Figure 5. The results show that by blending of SPL carbon material in anodes, the impurities (mainly Na) increase with the additions due to presence of bath material in SPL carbon. Other properties of anodes are not affected much.

Table 4. Properties of bench scale anodes.

Properties	**Normal anodes**	**Anodes with 4 % SPL carbon**	**Anodes with 2 % SPL carbon**	**Anodes with 1 % SPL carbon**	**Anodes with 0.5% SPL carbon**	**Anodes with 0.2 %SPL carbon**	**Anodes with 0.1 % SPL carbo**	**Anodes with 0%SPL carbon**
Green anode density g/cm^3	1.65–1.70	1.674	1.643	1.641	1.646	1.644	1.625	1.627
Baked anode density g/cm^3	1.50–1.60	1.580	1.55	1.555	1.554	1.563	1.535	1.529
% Air reactivity residue	65–85	67.55	63.29	66.5	74.45	67.59	67.7	68.95
%Carboxy reactivity residue	70–90	70.09	67.27	74.07	74.20	73.69	75.5	74.7
% Na	0.060 max	0.28	0.21	0.16	0.15	0.12	0.060	0.051
% F		0.027	0.018	0.012	0.010	0.010	0.009	0.007
% Fe	0.13 max	0.11	0.094	0.089	0.090	0.086	0.085	0.084
% Si	0.03 max	0.034	0.030	0.029	0.027	0.0267	0.0245	0.0266
% CN	Nil	Not detectable	Not detectable	Not detectable	Not detectable	Not detectable	Not detectable	Not detectable

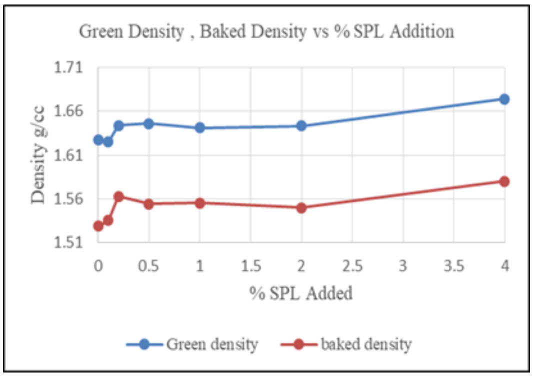

Figure 3. Anode density vs SPL carbon addition.

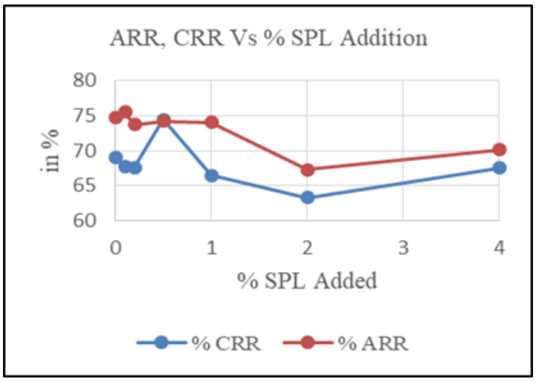

Figure 4. Anode reactivity vs SPL carbon addition.

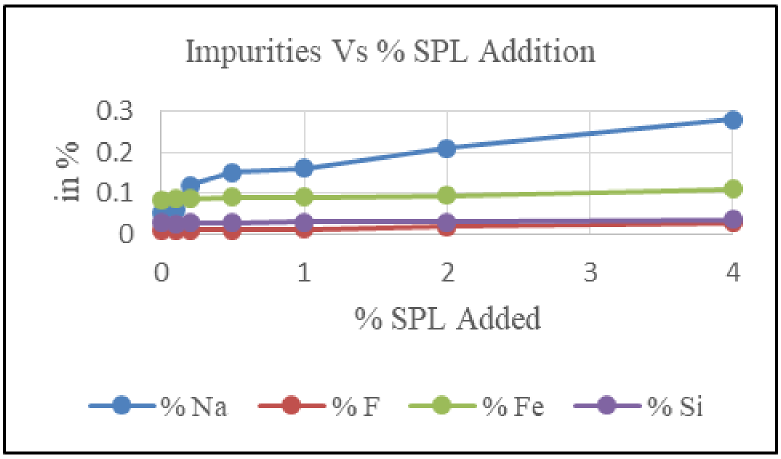

Figure 5. Anode impurities vs SPL carbon addition.

4.2. Phase-2 Experiments

Based on the findings of Experiment-1, second experiment was carried out to take out the leachable constituents of SPL carbon. The ground SPL was leached with water by keeping the solid to liquid ratio as 1:10 for a period of 24 h. The material was filtered and air dried. The residue was tested and results are tabulated in Table 5.

Table 5. Chemical composition of SPL.

Component	**Quantity (%)**
Carbon	70–75
Fe_2O_3	0.5–1
SiO_2	0.2–0.7
Na_2O	4–5
CaO	0.1–0.31
F	5–6
CN	0.07–0.08

By blending different amounts of this carbon material into the anode matrix, five batches of recipe were prepared with varied addition of ground SPL starting from 0.2 % addition up to 4 % addition in other baked butt fractions as shown in Table 6, for making bench scale anodes. Bench scale anodes were prepared as detailed at Table 6.

Table 6. Properties of bench scale anodes by addition room temperature water leaching.

Properties	**Normal anode**	**Anodes with 4 % SPL carbon**	**Anodes with 2 % SPL carbon**	**Anodes with 1 % SPL carbon**	**Anodes with 0.5 % SPL carbon**	**Anodes with 0.2 % SPL carbon**
Green anode density g/cm^3	1.65–1.70	1.669	1.655	1.641	1.646	1.644
Baked anode density g/cm^3	1.50–1.60	1.558	1.555	1.555	1.554	1.563
% Air reactivity residue	65–85	66.55	65.45	68.5	69.45	67.59

% Carboxy reactivity residue	70–90	71.09	72.5	73.07	72.66	73.69
% Na	0.060 max	0.152	0.058	0.050	0.045	0.040
% F		0.018	0.009	0.008	0.009	0.008
% Fe	0.13 max	0.107	0.094	0.0935	0.091	0.0856
% Si	0.03 max	0.0345	0.033	0.030	0.028	0.0278
% CN	Nil	Not detectable	Not detectable	Not detectable	Not detectable	Not detectable

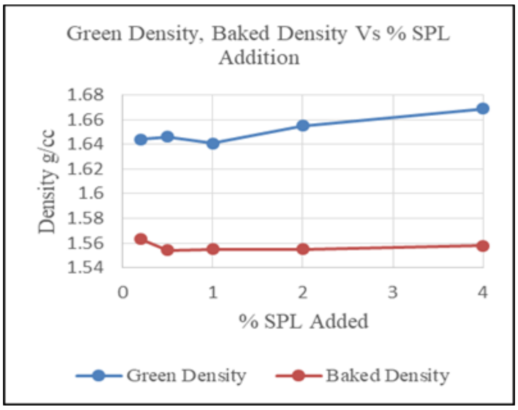

Figure 6. Anode density vs SPL carbon addition.

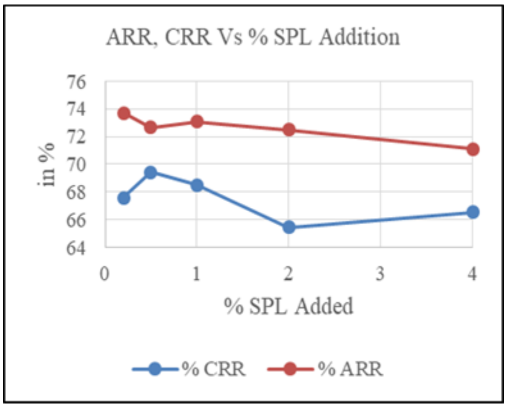

Figure 7. Anode reactivity vs SPL carbon addition.

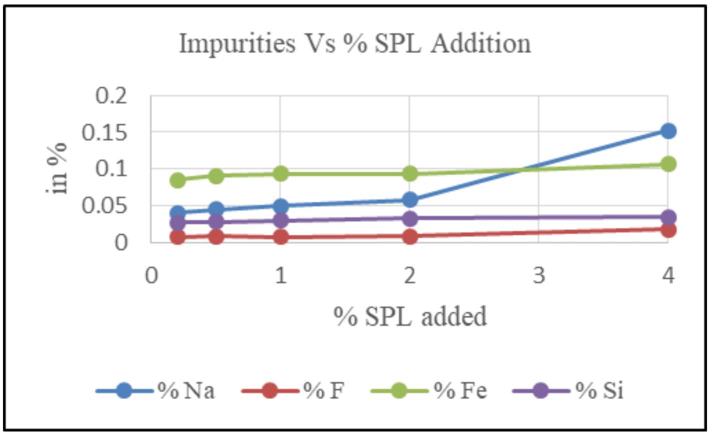

Figure 8. Anode impurities vs SPL carbon addition.

The filtrate obtained from Experiment-2 was tested. The pH of the liquor was 11–12 and the fluoride in water sample ranged from 2530–3710 ppm, the Na in water sample ranged from 2250–4210 ppm and CN in water sample ranged from 110–130 ppm.

The filtrate was treated with lime to recover the fluoride values as calcium fluoride. The purity of calcium fluoride was found to be 60–65 %.

Observations: By water leaching of the SPL carbon sample the leachable fluorides and cyanides come to the solution part. These contents in carbon residue decrease. Influence of SPL carbon (after leaching) additions on anode density is shown in Figure 6, on reactivity behavior is shown in Figure 7 and on impurities is shown in Figure 8.The bench scale results show that up to 1 % of SPL carbon can be safely blended into the anode carbon recipe without affecting the quality .The caustic solution can be discharged after neutralization or can be recovered after removal of fluoride for recycling in alumina refinery after increasing its concentration either by repeat recycling during leaching process or by evaporation.

4.3. Phase-3 Experiments

Based on the promising results obtained in phases 1 and 2, experiments were carried out to remove cyanide and fluoride from the liquid medium to recover caustic. Bench scale experiments were also carried out using the carbon residue in green anode recipe.

4.3.1. Pressure Leaching to Destruct Cyanide

Another batch of samples was used in the experiment ground SPL carbon sample 450 g with the chemical composition as given in Table 7 was added to 4500 L water and the material was taken in a pressure vessel 7.5 L capacity, Parr reactor. The experiments were carried out at 1.1 MPa pressure. Temperature noted was 180 °C. The material after autoclave leaching was left for cooling down and material was taken out at room temperature. The slurry was filtered and the residue and the filtrate were analyzed as shown in Table 7.

Figure 9. Parr pressure reactor 7.5 liter capacity

Table 7. Composition of high pressure leach solution residues.

Component	Raw SPL	Room temperature leaching 24 h	High pressure leached sample batch-1	High pressure leached sample batch-2
% Carbon (C)	50.7	67.6	66.20	65.8
% Na	10.29	3.53	4.14	4.21
% F	10.27	5.02	5.02	4.99
% Al	2.47	3.39	3.13	3.25
% Si	1.10	1.93	2.37	2.53

% Fe	0.17	0.31	0.36	0.39
% Ca	0.62	1.21	1.34	1.53
% S	0.12	0.13	0.14	0.14
CN in leach solution ppm	-	44	0.18	0.12
NaOH in gpl	-	9.6	12.4	12
pH of leach solution	-	12.02	12.15	12.23
Fluoride in leach solution ppm	-	6200	5340	5460

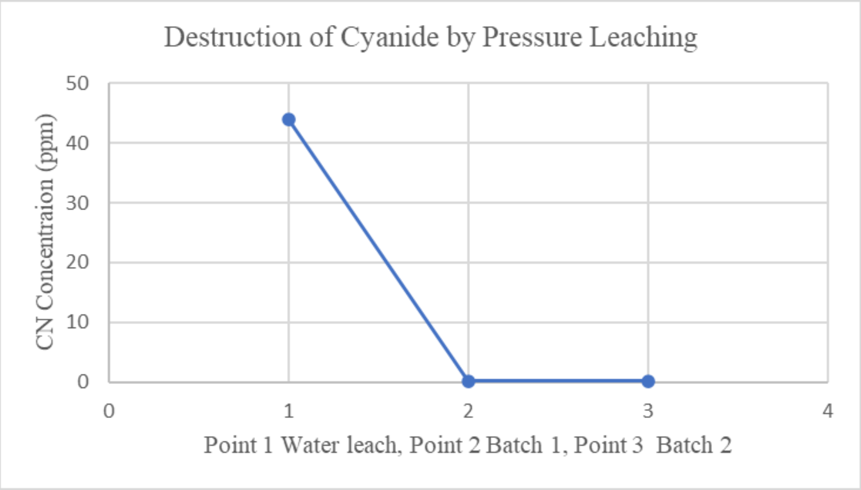

Figure 10. Destruction of cyanide by pressure leaching.

Observations:
It is noted from the above leaching experiment as shown at Figure 9, cyanide in the leach solution has decreased from 44 ppm to 0.12–0.18 ppm by pressure leaching.

4.3.2. Fluoride Removal by Liming the Liquor

Solution obtained from pressure leaching was subjected to lime treatment for removal fluoride as calcium fluoride precipitate.

$$Ca(OH)_2 + 2NaF = 2NaOH + CaF_2 \quad (9)$$
$$CaO + 2F = CaF_2 \quad (10)$$

Trial conditions and results are given in Table 8. Leach solution was found having 5460 ppm of initial fluoride concentration. Lime quantity was added as per stoichiometry. The solid precipitate after lime treatment was analyzed for detection of CaF_2 and confirmed using XRD and XRF.

Table 8. Lime treatment trials.

Trial No	Conditions	Final Fluoride in solution (ppm)
1	1 time lime, 24 h	992
2	1.3 times of lime, 24 h	520
3	2 times lime addition, 24 h	82
4	3 times lime addition, 24 h	75

4.3.3. Bench Scale Trials for Determining the Optimum Quantity of SPL Carbon in Anode Recipe

By blending different amounts of the carbon material obtained from phase 3 experiment, into the anode matrix, five batches of recipe were prepared with varied addition of ground SPL starting from 0 % addition up to 0.6 % addition in other baked butts fraction as shown in Table 8, for making bench scale anodes. Bench scale anodes were prepared as explained under phase1 experiment procedure.

Table 9. Anode properties developed by addition of high pressure leached SPL.

Properties	**Normal anode**	**Anodes with 0 % SPL carbon**	**Anodes with 0.1 % SPL carbon**	**Anodes with 0.2 % SPL carbon**	**Anodes with 0.4 % SPL carbon**	**Anodes with 0.6 % SPL carbon**
Green anode density g/cm^3	1.65–1.70	1.587	1.588	1.605	1.606	1.596
Baked anode density g/cm^3	1.50–1.60	1.491	1.489	1.508	1.509	1.496
% Air reactivity residue	65–85	77.787	78.122	75.561	79.289	75.155
% Carboxy reactivity residue	70–90	77.39	79.37	74.74	71.41	70.58
% Na	0.060 max	0.087	0.085	0.076	0.080	0.082
% Fe	0.13 max	0.102	0.113	0.10	0.092	0.126
% Si	0.03 max	0.030	0.027	0.025	0.020	0.029
% Ni	0.025max	0.017	0.018	0.018	0.014	0.019
% V	0.025 max	0.027	0.030	0.024	0.020	0.026
% S	3.00 max	2.56	2.61	2.3	2.3	2.31
% CN	Nil	Not detectable	Not detectable	Not detectable	Not detectable	Not detectable

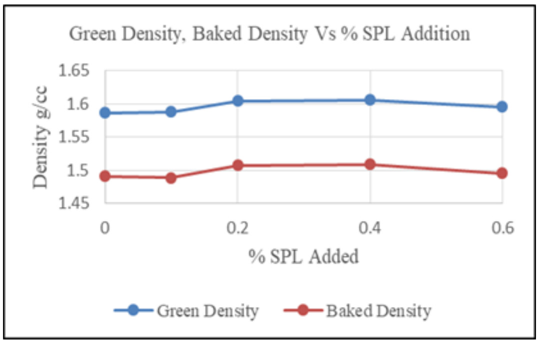

Figure 11. Anode density vs SPL carbon addition.

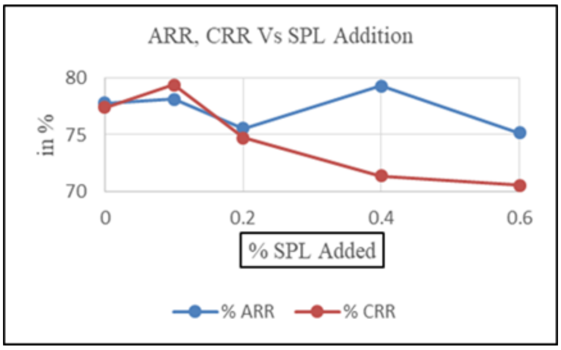

Figure 12. Anode reactivity vs SPL carbon addition.

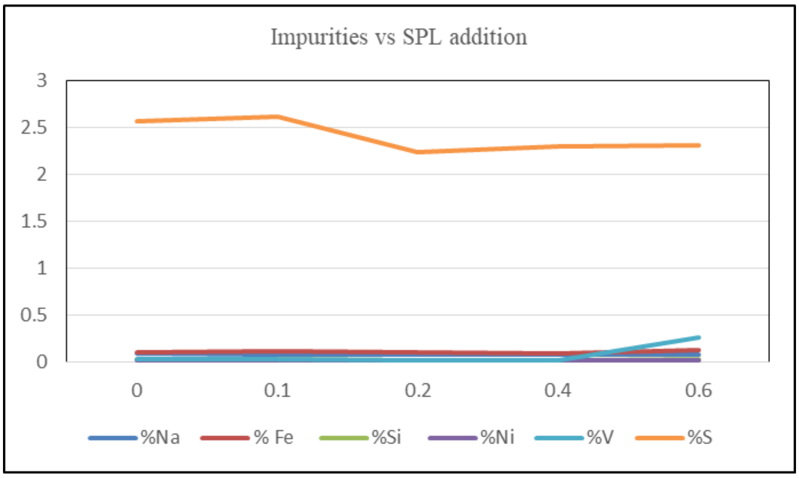

Figure 13. Anode impurities vs SPL carbon addition.

Observation: Influence of SPL Carbon (after pressure leaching) additions on anode density is shown in Figure 10, on reactivity behavior is shown in Figure 11 and on impurities is shown in Figure 12.The results of the above experiment show that minimal quantity of SPL carbon can be safely added into green anode recipe without affecting the quality of prebaked anodes. If 0.5 % SPL carbon is added to the anode recipe the quantity of SPL carbon that can be recycled per year in NALCO's smelter plant would be around 1500 t out of 2500-3000 t generated.

5. On-Going Work and Future Plan

i. Development of the process in 50-100 kg capacity and further optimization of leaching conditions, liquor recycling, fluoride recovery, NaOH quality, water balancing.
ii. Flow sheet development and pilot plant trial.
iii. Generation of sufficient quantity of material for validation of results in the plant.
iv. Large scale plant trial in limited pots with anodes made by addition of around 0.5 % SPL carbon obtained after leaching.

6. Conclusions

i. The composition of spent potlining material is widely variable depending on age of pots, digging procedure, storage and sampling.
ii. Preliminary lab scale studies conducted in the above study at NALCO show that it is feasible to reuse the carbon recovered from SPL carbon portion in pre baked anode recipe. The quantity to be recycled will depend on the % Na and % F in the material.
iii. The alkali extracted from the process can be recycled into the Bayer process after increasing its concentration.
iv. The non-hazardous calcium fluoride precipitated from the liquor can be sold to outside parties or can be disposed-off in cement plants.

7. References

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