

TREATMENT AND REUSE OF SPENT POT LINING, AN INDUSTRIAL APPLICATION IN A CEMENT KILN

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Abstract

The treatment of Spent Pot Lining (SPL) has been the occasion of numerous studies in the world. Several processes have reached the commercial stage in USA, Australia and Europe. Most of these processes generate waste that needs to be disposed of. An other approach is to make total use of the SPL constituents, with no waste disposal and with no air emission.

Among several processes allowing reuse of SPL, one is highlighted here : the addition of second cut (refractory) SPL to quarry materials in the raw feed of a Cement Kiln. Under close supervision of the French Environment Protection Authority, ALUMINIUM PECHINEY has developed a partnership with CIMENTS D'ORIGNY including an R&D programme to demonstrate the capability of a Cement Kiln to accept SPL as a raw material despite the presence of sodium and fluoride.

Since 1997, SPL are now recycled industrially in a Cement Plant of northern France.

1 - Definition of spent potlining

At the end of its life, the aluminium electrolysis pot has to be shut down to replace the cathode which is worn out and possibly damaged.

After removing as much bath and metal as possible and separating the cathode bars from the rest, the materials left are the Spent Potlining (SPL) comprising carbon materials (first cut) originating from the cathode blocks, side blocks and lining

paste and then the refractory materials (second cut). SPL are impregnated with fluoride and sodium salts and contain impurities, particularly traces of cyanides.

The composition of SPL is variable with the cyanides concentrated in the carbonaceous fraction near the cathode bars pot shell windows.

During the old cathode demolition, which might be carried out wet or dry, some smelters separate the carbon fraction from the refractory fraction depending on the solutions considered for SPL treatment.

2 - Frequent practices in the older smelters

In some smelters, Spent Potlinings are simply placed in a landfill or on the sea side where cyanides are partly and slowly decomposed by oxidation or ultra-violet light, and where the soluble fluoride salts are partly leached by the rain. In some cases, sealed surfaces (natural clay layers or artificial surfaces) allow the collection and treatment of leachates.

An alternative is to set up an accelerated leaching process, in a watertight pool where the crushed SPL are washed in closed circuits by large quantities of water with additives, such as bleach (sodium hypochlorite), to oxidise free cyanides. At the end of each washing cycle, the dissolved sodium fluorides are precipitated into calcium fluoride to be landfilled and the soda solution may be neutralised. This rather inexpensive process remains very attractive in the case of small smelters where the overall pollution potential is minor due to the small total quantities of residue in a single landfill.

3 - Some of the most advanced processes

During recent years, important research programmes have been carried out to treat spent potlining with the aim of producing an inert material or better to re-use some of the fractions of SPL. Some of the main processes having reached at least the pilot or prototype stage are described below.

3.1 - Alcan LCLL Process (Low Caustic Leaching and Liming) (1) (2)

It is a two stage hydrometallurgical process followed by a high pressure cyanide destruction. The by-products are sodium fluoride or calcium fluoride, a caustic solution and a solid residue which may be re-used in the cement industry.

The process developed on a pilot facility is being considered for the treatment of large quantities of spent potlining. ALCAN is studying the possibility of the construction in Quebec of a 60 000 t/year facility.

3.2 - Alcoa Australia Ausmelt Process (3) (4) (5) (19)

ALCOA Australia has developed a pyrometallurgical treatment at Portland with the Australian smelting technology company AUSMELT. A vitreous slag made of the mineral components of SPL is produced in a natural gas fired furnace. HF gas is released, cooled, filtered and subsequently passed through a reactor to produce aluminium fluoride.

The slag could be used as road base, as aggregate for concrete or if necessary landfilled. ALCOA Australia has carried out pilot and laboratory scale trials, and announced at the end of 1995 the construction of a 24 million AUD facility with a rated capacity of 12 000 tonnes of SPL per annum. This facility started up in 1998.

3.3 - Aluminium Pechiney Split Process (SPL Insolubilisation Technology) (6)

ALUMINIUM PECHINEY developed and tested the SPLIT process in 1991 and 1992 on a 3 000 t/year pilot facility installed at its research centre in Saint-Jean-de-Maurienne.

SPL are ground and then mixed with calcium sulphate before being introduced in a VICARB burner generating a hot air high velocity vortex. Cyanides are destroyed, fluorides are stabilised within seconds.

The gas/solid mixture is dry cooled and filtered through a bag filter. The solid residue is transformed into pellets to be landfilled.

ALUMINIUM PECHINEY has postponed its decision to invest in order to investigate and test various re-use solutions with other industries (Electric Steel and Cement Industries).

3.4 - Comalco Comtor Process (7) (8)

COMALCO is developing at Boyne Island a two stage process : cyanides are thermally destroyed in a TORBED reactor ; then fluoride and sodium are recovered in a hydrometallurgical treatment second step.

Early in 1990, COMALCO tested and operated a prototype calcination facility (5 000 t/year capacity), and since 1992 has operated a 10 000 t/year plant with a crushing facility and two calciners. A full scale caustic leaching plant is in operation since 1994.

3.5 - Elkem Pyrohydrolysis Process (9) (10) (11)

Carbonaceous ground SPL is heat treated in steam at a high temperature in a fluid bed reactor. Sodium aluminate, sodium oxide and soda are produced as well as gaseous HF. Aluminium fluoride is then synthesised.

10 000 tonnes of SPL were treated by ELKEM at Mosjoen during 5 years of operation, but the facility stopped for economic reasons in 1985. A similar process has also been developed and tested by KAISER ALUMINIUM, VAW and LURGI in a two year pilot plant programme at Lippewerk, Lünen, Germany.

3.6 - Elkem Electric Smelting Furnace Process (12) (13)

ELKEM has developed this process in a 100 kW electric furnace and through a pilot scale 600 kW electric smelting furnace bringing SPL together with iron ore and additives to a high temperature. The inert slag residue can be landfilled. The gaseous HF produced could be used to produce AlF_3 . This process seems to be comparable to the AUSMELT pyrometallurgical process but the energy is electricity instead of gas. ELKEM has not built a full scale industrial facility.

3.7 - Reynolds Metals Rotary Kiln Process (14) (15) (16) (20)

The carbonaceous and refractory fractions are crushed and mixed with limestone and brown sand (a calcium silicate waste from a sinter process alumina plant). The blend of SPL, limestone, and brown sand is fed to a rotary kiln where the cyanides are thermally destroyed. Soluble fluoride salts react with limestone to form insoluble calcium fluoride.

The REYNOLDS process was developed while treating over 300 000 tonnes of SPL between March 1988 and June 1990 in the Hurricane Creek alumina plant in Arkansas.

A commercial plant has been built at Gum Springs, Arkansas at a cost of 60 million USD with a capacity of 109 000 t/yr of SPL. Since start-up in October 1993 and through May 1995, 70 000 tonnes of SPL from 16 smelters in the USA and Canada have been processed.

The by-product of the REYNOLDS process was delisted from the list of hazardous waste by the US EPA on December 30, 1991.

However, it was discovered in the fall of 1996, that the leachate being generated from actual disposal of the treatment residues was more hazardous than initially anticipated : the treatment residues are piled in a dedicated landfill where the pH is alkaline, leading to fluoride concentrations much higher than the treatment standards. REYNOLDS has agreed to give up the delisting, and the administrative process of withdrawing the delisting has been undertaken.

While treatment of fluoride is an important indicator, the EPA views cyanide reduction as more important, and concludes that the REYNOLDS process provides substantial treatment. Due to the existence of the REYNOLDS facility treatment capacity, the EPA has, under the Land Disposal Restrictions (LDR) program, prohibited SPL to be landfilled after October 8, 1997 without being treated to meet LDR standards which are based upon the performance demonstrated by the REYNOLDS process.

3.8 - Vortec Vitrification Process (17)

In 1995, ORMET PRIMARY ALUMINIUM CORPORATION purchased VORTEC's patented CMS vitrification process to treat SPL waste and started operation at Hannibal, Ohio in 1996. The facility is designed to treat about 25 tonnes per day of SPL, and produce a glass frit intended to be used in glass and ceramic manufacturing operations. In July 1997, the US EPA and Quebec's « Ministère de l'Environnement et de la Faune » (MEF) have approved this process to make glass products from SPL.

In November 1997 VORTEC and ORMET formed a joint technology development enterprise « SPL Recycling ». VORTEC has also strategic alliances with HOOGOVENS TECHNICAL SERVICES, a supplier of gas treatment facilities for the aluminium industry, and with WELCO INDUSTRIALE (Italy), a ceramic tile manufacturing equipment supplier. New SPL treatment projects are currently being evaluated.

3.9 - Production of Cryolite (18)

Industrial production of synthetic cryolite has been carried out in Germany since 1950 in a former alumina refinery presently producing 7 000 to 10 000 t/yr of cryolite, from 15 000 to 20 000 t/yr of SPL. The facility has a capacity of treating 40 000 t/yr of SPL. Although cryolite consumption is decreasing in the aluminium industry, the cryolite market still seems to be attractive.

The plant, as operated by RETHMANN LIPPEWERK, uses a fully hydrometallurgical process. The final solid residue is washed and may be used humid as a secondary fuel cement kilns.

4 - Some limitations to the treatment processes

The leachability of fluorides still contained in the residue after a given SPL treatment is a major factor for the evaluation of the performance of the process.

A specific characteristic of the fluoride ion is that its leachability is highly pH dependent. It remains low for moderately acidic, neutral or moderately alkaline conditions, especially when calcium is present to form CaF_2 . But F solubility increases drastically in a more acidic or alkaline environment.

Of course, this may vary according to the buffering effect of other ions present, but generally speaking, the solubility curve is similar to the one presented in figure 4.1 below.

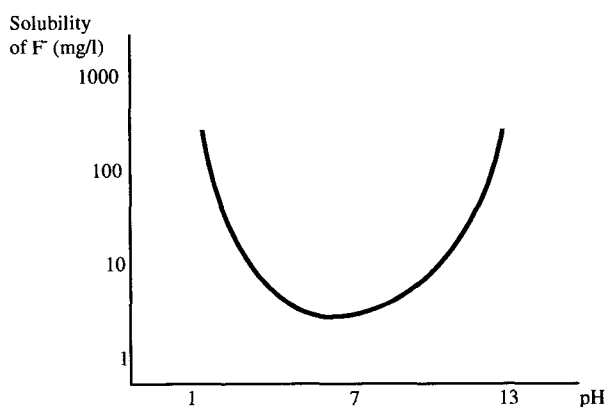


Figure 4.1: Example of solubility curve of fluorides, as a function of pH

This will of course have to be taken into account when evaluating the performance of a SPL treatment process. Knowing the remaining alkalinity of the treatment residue as well as its stability with time will allow evaluating the long term performance of the process.

Another factor to take into account is the chemistry of the leaching test. Two examples may be compared : the TCLP test as recommended by the US EPA and in the French NF leaching test. The following table allows visualising their differences.

Table I: TCLP (US) and NF (France) leaching test

	TCLP (US)	NF (France)
Sample preparation, grinding to:	< 9.5 mm	< 4 mm
Leaching solution	acetic acid	distilled water
Number of leachings, duration	one single leaching, 18 h ± 2 hours	3 consecutive leachings, 16 hours each

Knowing the curve presented above, it is then interesting to see what will be the effect of the leaching test on a process evaluation. For example, a treatment process which leaves a high free alkalinity in the residue may give satisfactory results for leachable fluorides according to the US procedure because of the buffering combination of acetic acid + sample alkalinity. But poor performance may be seen when applying a French leaching test on the same sample where the leachate pH will be free to increase with the alkalinity of the residue. In some cases such phenomenon were found to be responsible for poor performance of a SPL treatment process.

5 - Industrial re-use of spent potlining

Concurrently to the development of treatment processes leading to landfilling of a non-hazardous waste, it has been a practice to try to re-use economically, in other processes, all or a fraction of SPL when possible. Due to the foreseen closure of a large number of private landfills and the expected high future fees for landfilling into external landfills, the re-use route with other industries is even more attractive today.

However, legal procedures to obtain an operating licence for industrial testing, transport, crushing, and re-use can be long and difficult. And then, the re-use by another industry has to be profitable for both parties.

The use of SPL in other processes will often be made easier if various SPL fractions are carefully sorted out.

5.1 - Re-Use of SPL in Electric Steel Furnaces

Modern electric steel furnaces (approx. 100 tonnes capacity) are used to melt scrap steel very quickly. Approximately 45 minutes are needed per load. The steel is then poured into a ladle furnace where metallurgical adjustments are carried out.

Normal operation in the furnace is carried out with an addition of lime (CaO, approx. 3,5 kg/t) and carbon (anthracite, approx. 12 kg/t, particle size approx. 10 mm) added to the scrap steel. During the last 10 minutes of each furnace operation, fine petroleum coke particles are injected along with oxygen into the slag to cover the electric arc with foam and allow higher power without damage to the furnace lining.

After the liquid steel has been poured into a ladle furnace, normal operation includes the addition of lime ground to a smaller particle size than in the smelting furnace, in order to rapidly produce a new slag. Other additives are used too : desoxidation agents such as aluminium, metal alloys to meet the final composition, fluorspar (approx. 0.6 kg/t of steel), and alumina to fluidify the slag.

Replacing that last addition of fluorspar by the carbon fraction of SPL has been tested in the U.S. where it was found to improve the fluidity of slags and reduce the sulphur and phosphorus levels. Large scale successful testing is also believed to have taken place in Russia.

In Western Europe, the Italian electric steel industry has a tradition of re-using carbonaceous SPL. The Italian legislation is particularly favourable : a general law enacted by the Ministry of Environment on the re-use of residues from a production cycle into another production or combustion process, authorises specifically the re-use of carbonaceous cathodic residues from the demolition of aluminium electrolytic cells in electric furnaces for the production of steel as a fluidification and desoxidation agent. Since May 1st, 1994, the transfer from another European Union country or from an OECD country must comply with the European Council regulation 259/93 of February 1st, 1993 (Amber list of wastes).

Carbonaceous SPL is generally crushed to 15/50 mm or 15/80 mm and mixed with fossil carbon in specified proportions before being used in the electric furnaces. Finer particles are not preferred as much : some fine particles (0.6/2 mm) are also used mixed with petroleum coke for the foaming slag process. Some are agglomerated with molasses or starch and used in the pouring process (ladle furnace). SPL from dry delining are preferred.

The use of SPL in the electric steel industry is a very attractive method because the cyanides are destroyed by the temperature. Carbon as well as fluorides are already used by this industry, although it is common practice to use less and less fluorspar in order to save the linings of the ladle furnaces.

Some selected fractions of refractory SPL, rich in alumina, can also be used after mixing with alumina to reach a content larger than 60 % in Al₂O₃, as a fluidification agent in the ladle furnace. Refractory SPL crushed and mixed with a suitable concrete is also used in some electric furnaces to repair the lining.

Most of the carbonaceous additives using SPL are used in electric steel furnaces located less than 200 km from the crushing/mixing facilities.

5.2 - Re-Use of SPL in the Cement Industry

Re-use of SPL is possible in the cement industry. Two options have been considered : either the carbon fraction can be used for its fuel value, or the refractory fraction can be used as a raw material substitute.

In both cases, one has to carefully consider the sodium and the fluoride balances of the cement kiln where SPL may be recycled. Those two elements are commonly present in the raw materials coming from the clay and limestone quarries. The proportion of SPL accepted by the process will depend mainly on the proportions of sodium and fluoride already present in the process, since there is a limit for both of them in the cement. This evaluation will have to be addressed for each plant where SPL is thought to be recycled.

Both carbon and refractory SPL were tested as supplements in the cement manufacturing industries, and routine use is now underway since 1997 in one kiln in France after a long term evaluation programme which is presented here (§6).

5.3.1 - The carbon fraction. Extensive test work was carried out in the US during the early 1980s and concluded that well sorted carbon fractions of SPL could be blended with the coal (more than 2 % of SPL in the mixture) without any negative effect on the process. The stack emissions and the quality of the clinker produced during that test work were not affected by the presence of SPL.

Following that test work, some time was necessary to evaluate the legal frame and the type of operating licence necessary for a routine recycling activity.

In 1988, things were considerably slowed down when SPL were listed as a hazardous waste by the US EPA.

5.3.2 - The refractory fraction. The presence of alumina and silica in SPL allows another approach in recycling. Those two elements are present in the quarry materials in proportions which may be not sufficient. In some plants, an addition of alumina may even be necessary.

Adding a small proportion of well sorted refractory SPL to the raw material fed to the primary crusher was tested recently in France during a 2 year evaluation programme. This was done under the supervision of the Environment Protection Authorities, within the frame of a temporary operating licence followed by a permanent one, after it was demonstrated that no detectable effect could be found either at the stack or in the quality of the cement. The next chapter presents how this programme was accomplished.

6 - Cement kiln reuse qualification programme

The capacity of the Cement industry to process various industrial wastes is well known. However, there was no previous experience in France with SPL except for some test work performed in the late 1980s on the carbonaceous fraction quickly abandoned after some serious difficulties were encountered in the grinding circuits.

During 1994, preliminary contacts were established by the local Environment Protection Authorities between ALUMINIUM DUNKERQUE (a subsidiary of ALUMINIUM PECHINEY) and CIMENTS D'ORIGNY (a subsidiary of the HOLDERBANK Group), both located in northern France, 40 km apart.

The Wet Process used by the Lumbres ORIGNY Plant was found to be more adapted to the refractory fraction of SPL. A complete qualification programme was then established within the frame of a triangular partnership between ORIGNY, ALUMINIUM PECHINEY and the local EPA. Over two years were necessary to carry out the 4 main steps of that programme.

6.1 - Preliminary laboratory work

The purpose of that laboratory work was to determine the maximum allowable percentage of SPL which can be added to the raw feed of the kiln on the basis of the following parameters :

- acceptable concentration of F and Na in the final clinker,
- rheology of the raw feed,
- behaviour of F and Na in the kiln.

Taking into account the pre-existing levels of alkaline compounds in the quarry materials, it was found that the addition of refractory SPL should not be higher than 0.6 %.

6.2 - First industrial test

The second step was carried out in order to study the behaviour of SPL in the feed preparation circuit of the raw paste.

60 tonnes of uncrushed SPL were fed regularly to the primary crusher at a controlled rate of 0.6 %. At the same time, much attention was paid to the rheological characteristics of the paste. CN, Na and F analyses were performed on the main flows including the closed loop make up water of the mills.

No noticeable effects were found. The decision to go ahead with a long term test was taken in order to study the effect of SPL on all the circuits, including the kiln, and to accumulate stack emission data over a duration of at least one month.

6.2 - Temporary operating licence

An application was prepared early in 1995 and then filed during the month of July 1995. The authorisation to process 400 tonnes of SPL over a period of 6 weeks was granted early in 1996 after a full investigation by the Authorities - Industry Ministry, Agriculture Ministry, Public Health Organisations and Land Management Offices.

6.3 - Full size 400 t trial

During 4 weeks, refractory SPL were fed to the primary crusher at a constant rate of 0.3 % and then for an other 2 weeks the proportion was increased up to 0.55 %. Then all of the monitoring programme set during that test was continued for an extra week, 2 weeks after the end of the test in order to monitor the circuits and flows in a non SPL situation.

Over 800 samples were taken and analysed during those two months.

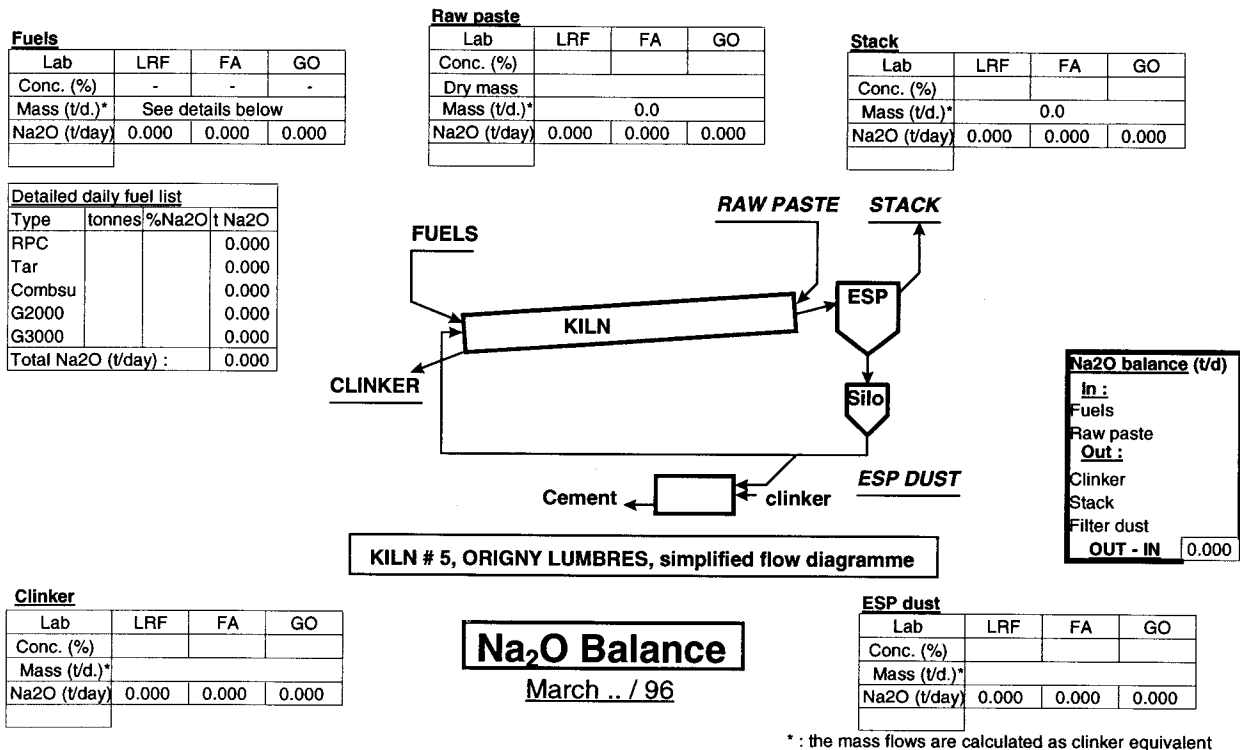


Figure 6.1: Example of daily mass balance

6.3.1 - Crushing, grinding and paste preparation. As during the first 60 t test, no problem was encountered with the crushing and milling circuits.

The concentration of total cyanides remained below the detection limit in all the waters of the plant except in the wet mill itself where the water CNt concentration went from < 0.008 mg/l up to 0.04 mg/l at the most. The fluoride levels in the process water followed the concentration of SPL, but never exceeded 8 mg/l. The quality of the excess water rejected to the river was not affected at all.

6.3.2 - Kiln circuits. On a daily basis, and for 44 days, a complete mass balance was computed on the basis of Na and F analysis performed each day on all the flows. A daily table such as the one presented in figure 6.1 was then established

One of the main goals of the material balance was to closely follow the risk of accumulation of Na and F in the kiln. A positive material balance indicates an excess, a negative one means that some Na or F is retained in the kiln.

The results showed that the risk did not actually exist. For a period of 44 days, a total quantity of 134 tonnes of Na and 51 tonnes of F were fed into the kiln from all sources and both cumulative material balances closed at almost zero (Na at + 2 % and F at + 0.9 %).

On a daily basis, the only fluctuations encountered were due to stock level variations. Figures 6.2 and 6.3 present the sodium and fluoride balances of each day.

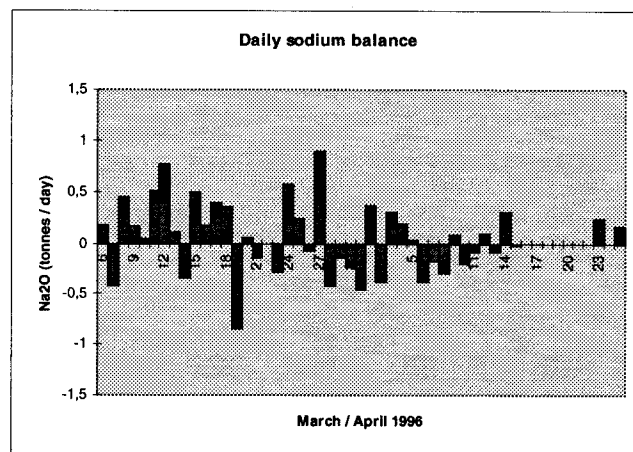


Figure 6.2: Daily sodium balance

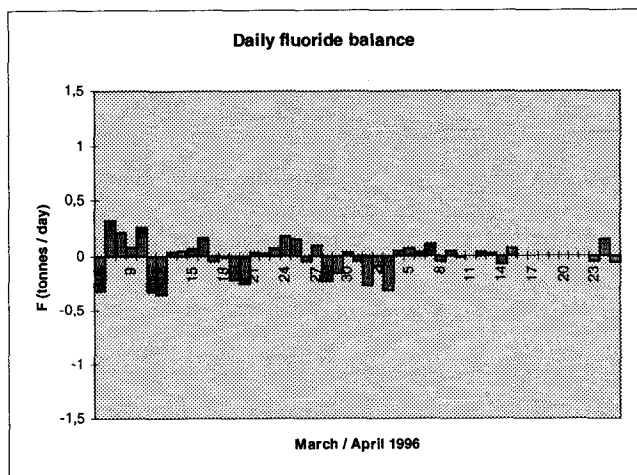


Figure 6.3: Daily fluoride balance

6.3.3 - Stack monitoring. Over the test period, the stack was sampled 17 times, and the gasses were analysed for HF, particulate fluorides and cyanides.

Figure 6.4 is a summary of the results for fluorides. It shows clearly that there was no noticeable effect on the gas quality.

These results were confirmed by an independent laboratory who monitored the stack at the same time ALUMINIUM PECHINEY was doing it. On March 21st, an HF concentration of 0.30 mg / Nm³ of wet gas was found at the stack.

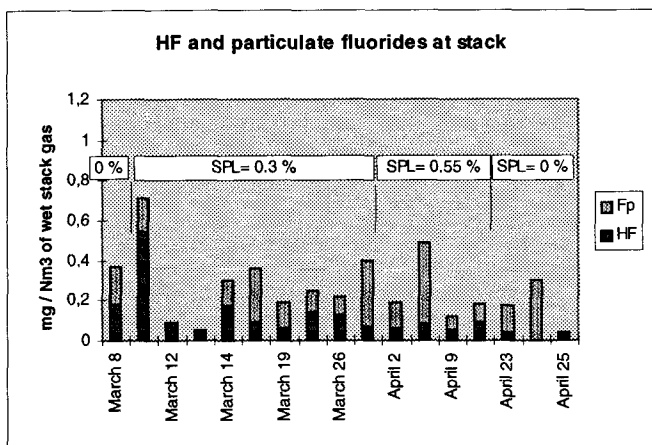


Figure 6.4: F stack emissions

As far as the cyanides are concerned, 8 samplings and analyses were performed. No result exceeded the detection limit, < 0.0008 mg / Nm³, for both gaseous HCN and particulate total CN.

6.3.4 - Cement quality. An extensive quality control programme was carried out on three different cement samples. A typical « 0 % SPL » one, a « 0.3 % » and a « 0.55 % » and three concrete samples made from the same 3 cement samples were submitted to 2 types of tests.

After a triple leaching test (3 x 16 hours consecutively, on a ground fraction) according to NF standard NFX 31210, the 6 samples were analysed for soluble F, Na and CN.

Cyanides were never detected. F and Na did not change at all with the presence of SPL, neither on the cement, nor on the concrete.

Compression strength was tested after 2 days and after 28 days of setting on normalised concrete samples. The presence of SPL had no effect on that parameter.

6.3.5 - Environment control. Around the city of Lumbres, an existing network of static samplers allow the monitoring of the air quality several kilometres downwind from the cement plant.

Particular attention was given to the analytical results and the frequency of the sampling was increased during the test period, in order to accumulate comparable data, with and without the presence of SPL in the kiln feed.

It was found that Na, F and CN concentrations in the sedimented dust were not affected during the test period. They were even found lower than normal.

6.4 - Permanent valorisation

The conclusions of the 400-tonne trial were found positive in all aspects. After submission of a detailed exhaustive report to the EPA authorities, the Cement Process of the Lumbres plant was declared suitable for the total reuse of refractory SPL.

However, since the operating licence of the plant had to be modified to include SPL, a new application for permanent operation had to be filed. The results of the test helped to expedite the procedure and the authorisation came quickly.

Since 1997, addition of refractory SPL is now a routine operation at Lumbres ORIGNY Plant.

7 - Future trends and conclusions

A thorough qualification programme applied to a process known for its capacity to reuse wastes successfully allowed opening a new door in the field of SPL valorisation.

This 2 year partnership between CEMENTS D'ORIGNY and ALUMINIUM PECHINEY is now becoming a reference in France. The Administration has played a key role in this development and word is spreading : what was done in northern France can be done in southern France.

The future developments are then very clear : each smelter is now engaged in an inventory of all the cement kilns in its vicinity, and of the pre-existing levels of sodium and fluorides in the clinkers produced.

A new qualification programme is already underway in southern France on a different cement fabrication process. The local EPA authorities, perfectly aware of the results obtained in northern France follow closely and with evident interest the development of the tests.

We have not yet reached our goal to recycle all the refractory SPL produced by the French smelters through the cement industry, but we are coming closer every day !

In the near future, a similar approach will be confirmed for the carbon fraction of SPL. However, this may need a more careful approach, since it is not yet a tradition for the steel industry to act as a waste cleaner...

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