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**STABILISATION OF HEAVY METAL  
FROM AUTOMOBILE GRINDING RESIDUES.**

**BENEFICIAL REUSE OF THE RESIDUES.**

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## ***INTRODUCTION.***

For decades some 75 per cent by weight of an End-of Life-Vehicle (ELV) have been recycled, and currently go mainly to landfill sites. This portion of the vehicle is made up more or less equally of inorganic and organic substances. The organic substances are mainly plastic materials followed by a small quantity of modified natural substances such as cellulose fiber products or leather. Metallic materials from ELVs are relatively easy to recycle without major input of costly manual labour. The separation of ferromagnetic materials from other metallic materials is also easy and relatively inexpensive.

Since the techniques for recycling metals are targeted specifically at their material properties, metals can only be recycled as metallic materials although it is often necessary to subsequently produce other alloys from these metals to enable their use in alternative product applications. However, recycling of automobile using grinding process generates also fines particles which contains plastic and metallic materials like Chromium, lead, iron, copper and zinc among which some are toxic at low concentration. There are not many process available for the treatment of these contaminated fines particles which is landfilled at this time. We are developed adequate processing methods to transform metal ion from automobile grinding residues fines particles (AGRFP) into new products for beneficial reuse using phosphates. Minerals can be recycled if they are non-hazardous. Heavy metal stabilisation, i.e. converting trace metal ions into insoluble, stable and non-leachable compounds is a key step for our process. We feel the issue goes beyond incorporating the minerals in a properly selected matrix and is well worth considering specific treatment methods in order to obtain desired properties.

The process use is based on stabilisation of heavy metal using phosphates. In fact, Solvay have recently developed a new treatment uses phosphoric acid to neutralise fly ash basicity and form insoluble metal phosphates. It is composed of four parts: fly ash is washed and filtered to extract soluble salts (mostly chlorides) before chemical treatment with phosphoric acid. Thermal treatment transforms the solid into a stable mineral phase. In order to engineer the final product to have the desired properties for end use, we studied the control of the physico-chemical properties by specific thermal

treatments. This example was applied in REVASOL experimental project leading to the reuse of stabilised fly ash in road construction.

The calcination of the chemically treated waste gives solid residues containing apatite mineral-like insoluble metal phosphates without generating volatile metal chlorides and oxides. Calcination significantly affects the solubility of the metals ions. The treatment under mild conditions contributes to apatite crystallisation. Such transformed material with stabilised trace metal ions may be considered for beneficial reuse. In fact, These apatite containing solids are stable residues which may be added to various materials to facilitate recycling and beneficial use in highway, concrete or cement products.

Eliminating disposal sites and transforming hazardous materials into new products for beneficial reuse is an investment for a better future. Sustainable growth in the automobile recycling area will only be possible with recycling of the AGRFP.

The paper will focused on the review of the literature concerning the treatment of AGR and on an example showing how chemical treatment with phosphates and thermal treatment (calcination) can modify several solid waste containing heavy metals and transform its components into environmentally friendly products or natural resources conservation products. In this paper, the incineration fly ash will be taken as an example of valorisation of solid waste containing heavy metals.

## ***1 BECOMING OF AN ELV.***

### *1.1 Automobile Waste: « zero tip » [1].*

Every year, in France, 1.8 million of End-of-Life Vehicles (ELVs) go for scrap. These wrecks, that weigh a ton on average each, are recycled to 75% (essentially metals), which places the car among the more recycled products of current utilisation. The 25% remaining, about 500 000 tons of wastes per year, are Grinding Residues (GR) which are a mixture of glass, rubber, plastic, paint, leather and textile. They generally end up in a rubbish dump, whereas a big part could be valorised.

Automobile Grinding Residues (AGR) are residues of residues. Indeed, after grinding of carcasses and separation of ferrous and non-ferrous metals, one gets a residual material composed of mousses, plastic, glass, rubber, metals and minerals. This waste deposit is

estimated to 500 000 tons per year. Of the point of view of the rules, the AGRs are assimilated to Banal Industrial Waste (BIW). It means that they must be re-used or recycled as materials or energy sources (law of July 13, 1992). The landfilling of AGRs is prohibited without treatment.

Many ways of valorisation of AGRs are available, especially since the agreement-setting of March 10, 1993, fixing the objective of valorisation at 85% of the ELVs in 2002 and obliging to valorise at least 40% of the AGRs. The thermal valorisation (by incineration) represents a main way of reprocessing.

### *1.2 The ideal recycling [2].*

Ideally, a leading recycling center, where all activities are regrouped in only one site, is able to reprocess until 95% of the car weight.

The pilot recycling centers process about 220 000 ELVs per year. They allow the recuperation of metals, glasses, tires, liquids, and of some plastic materials. Residues are largely used as combustible in cement factory. Technically, all operates, stay only one obstacle to the generalisation of such pilot centers: the absence of real industrial paths to use the recuperated materials and to make of the recycling a profitable business. The agreement, signed in 1993 by constructors, the authorities and actors of the recycling, that set themselves the target to recycle 85% of the car weight in 2002, and 95% toward 2015, specify indeed that “these objectives will only be achieved if this path finds by itself its economic balance”. Excepted these two pilot centers, the majority of cars is processed by the traditional way. Breakers disassemble what is most profitable to resell: aluminium, copper and mechanical pieces. Most serious among them, a minority, clean up the cars. For lack of industrial paths to recycle other materials, they are content with recovering the ferrous and non-ferrous metals (that represent more than 70% of the car weight all the same), the remainder going to the tip.

- **Cleaning up**

This operation consists in emptying the car of all its liquids (oil, liquids of windscreen, cooling and brakes) and to remove the battery. Tires are also disassembled. This first step is indispensable. Indeed, a car non-cleaned up will corrupt the quality of the AGRs destined to the tip. These residues, in this state, will be therefore a supplementary pollution source. Oil is recycled or used as combustible in cement factory. The usable

tires are retreaded, others are ground to serve as combustible in cement factory. Plastic (polypropylene) and the lead of batteries are reused as raw materials.

- Disassembly

The glass is recuperated. For plastic, the disassembly limits itself for the meantime to polyurethane foams of seat and polypropylenes, mainly present in shields. Some non-ferrous metals, as the aluminium or the copper, are recuperated also. The catalytic exhaust pipes are disassembled in the goal to collect the rare metals that they contain (platinum and rhodium). Some mechanical pieces, as motors, are got back before being renovated and sold second-hand. After treatment, the glass is transformed in fiberglass, bottles or food packings. Foams of seats are recycled in soundproofing and insulating. And shields are useful to manufacture new shields. Non-ferrous metals are melted and reused as raw materials.

- Sorting

Wires of copper, previously disassembled, are stripped in a separated workshop. It's a manual operation, justified by the price of this raw material.

- Grinding

The grinder tears carcasses of cars to shreds. Begin the different steps of the automatic sorting then: a magnetic pulley separates the ferrous metals destined to be remolded; a system of blowers separates the slight wastes (foams and shreds of plastic) good for the tip; an electric current separates non-ferrous metals of the thickest pieces of plastic; a bed of water finally separates non-ferrous metals the some of others, the heavy (zinc, lead, copper) remaining under water and slightest (aluminium, magnesium) coming back up to the surface. Grinding residues are to their turn sorted out in two parts: the organic residues (plastic, rubber) usable as combustible in ovens of cement factory, and the mineral, incombustible residues, that leave to the tip.

Between 90 and 95% of the weight of a car has been recycled, against 75% in a classic treatment limiting itself to the recuperation of metals. In the assumption of a reuse of the mineral grinding residues, to do embankments for example, 100% will even be reached. Cars will be then completely recyclable.

## 2 VALORISATION PROCESSES OF ELVS.

### 2.1 Incineration of wastes.

According to their origins, the incinerated wastes bring energy, according to their Lower Calorific Value (LCV), interesting physical elements for the manufacturing of the cement (wastes of iron oxide of the metallurgy, silica of foundry sands, alumina...). The heavy metal rejection generates an important problem however most of heavy metals are toxic and are not destroyed during the combustion process. They undergo in fact simple chemical transformations.

#### 2.1.1 Valerco and Vicat: Residues are used as combustible.

The automobile grinding site at Saint-Pierre-de-Chandieu in France, prefigure the complete path of the automobile recycling. Several units of the “French Company of scrap metals” (CFF) are established there: the grinding Purfer, the demolition worker Ecoval, specialist of the cleaning up and the disassembly, created in partnership with PSA.

In fact Valerco is the results of a partnership between the CFF and the Vicat cement factory, valorise about 15 000 tons of AGRs per year. Created in 1991, the Valerco society has for mission to prepare the combustible stem from grinding residues (provide by the grinding Purfer). This combustible is destined to be burnt in ovens of the Vicat cement factory, installed at Montallieu, in Isère (France). On the 30 000 tons of AGRs that Saint-Pierre-de-Chandieu generates every year, only half enters in the composition of the combustible.

Indeed, after a first sifting of residues, close to 15 000 tons appear under form of “fine”, mainly organised by minerals (silica, calcium salt, oxide of iron). These wastes are destined to the tip. Remain 15 000 valorisable tons organised of moss, rubber and various plastics. It is on this material that Valerco operates, pulling to pieces it with the help of a grinder with knife to form tablets of fissile material of a 6 cm maximum diameter. A new technique for product preparation allowed a significant lowering of

their chlorine rate; the faculty to the burning thus improved allows to offer other openings.

To burn the AGRs, the Vicat cement factory installed a system of precalcination, allowing to destroy all components of the AGRs, including heaviest. “We are today to consider the AGRs like a classical combustible”, confirm Raoul de Parisot, Director of the cement branch at Vicat. Thanks to the incineration of the AGRs, the site of Saint-Pierre-de-Chandieu valorises End-of-Life Vehicles to height of 87%.

### 2.1.2 Cement industry and the stabilisation of wastes [3].

In the same way, the Research and Development department of the Vicat group developed new cements for concretes with high resistance. Vicat developed in 2001 a binding of stabilisation (LIV) to reply the requests of the market of waste stabilisation (stabilisation of the municipal solid waste incineration fly ash).

## 2.2 *The thermolyse process.*

The incineration consists in eliminating waste by burning it (temperature superior to 850°C) with air atmosphere, while the thermolyse consists in decomposing waste by heating in poor air atmosphere at temperatures from 450 to 750°C.

The thermolyse certainly has a place to take among techniques of waste treatment.

The thermolyse does not constitute a total treatment of wastes, but a partial treatment that emerges on a solid-fuel. The thermolyse must be designed therefore upstream of an existing industrial unit able to use this combustible.

The residue of thermolyse is a carboned residue that is under the form of a homogeneous black coal powder, constituted of elements of a few millimeters. Its calorific value evidently depends on the incoming wastes. The residue stem from standard domestic wastes has a calorific value of 18/20 MJ/kg, that situates it in the poor ashy coal category.

This combustible presents advantages that it is a combustible of renewable substitution endlessly, with a significant calorific value, and with possibilities of consumption spread out in the time, since it can be stocked before a future utilisation, correlating to a peak of consumption.

The solid-fuel can be interesting for high energy consumer industries by increasing the rates of substitution of their combustible. Utilisations in metallurgy and in power station are in progress. However utilisations in cement factory constitute the best way today.

The important fact is to recall that the utilisation in cement factory would suppress all ultimate solid residues while incorporating the mineral materials contained in coke in components of the cement.

	Domestic wastes	Tires	AGRs	Plastic	Muds
Gas (kg/ton)	480	560	450	680	660
Potential energising LCV (MJ/kg)	17	40	12	28	n.p.
Semi-coke (kg/ton)	300	330	300	280	340
Metals / inert (kg/ton)	100:120	150	100:150	40	-
Potential energising of the semi-coke (MJ/kg)	18	29	7	5	7

*Table 1 : Balance material and LCV (LCV of 1 kg of coal = 30 MJ/kg, LCV of 1 kg of fuel-oil = 38 MJ/kg) according to wastes treaties in thermolyse (Source: Free University of Brussels).*

The washing of the solid stem from thermolyse allows to eliminate an important fraction of chlorine, inerts and metals.

The implantation of a thermolyse unit should be situated ideally close to a fossil energy user (valorisation of the thermolyse gases). The thermolyse solid-fuel is used ideally as combustible of substitution in cement factory.

Temperatures of treatment being weak, and the thermolyse being realised without oxygen, the heavy metals are not oxidised, nor volatilised (to the exception of mercury, and of cadmium whose emissions are lower to norms), what facilitates their recuperation and their elimination.

Thermolyse only generates very little pollution. First of all, since there is not combustion of wastes, but only combustion of gas, the volume of smokes is reduced (decreases of about 50% with regard to the incineration). But, especially, the system allows a perfect chlorine neutralisation by washing the carboned solid.

The ferrous and non-ferrous metals in output of thermolyse have an incomparable quality with the one of metals in output of incineration (it is not mixed to wastes), and even better than the one of metals recuperated in center of sorting, as they are rided of all complementary element (plastic, painting, water...). The price of trade-in should be elevated therefore. It is only about potential revenue that depends on the recuperation (magnetic current, current of Foucault...). It is able therefore to be interesting to mix treated wastes with the DIBS in order to increase valorisable fraction and to justify a complementary sorting plant.

### **3 TREATMENT OF INCINERATOR FLY ASH WITH PHOSPHORIC ACID AND VALORISATION [4, 5, 6, 7, 8].**

The fly ash particle size distribution (mean size), determined with a Mastersizer laser granulometer (Malvern), is 24  $\mu\text{m}$ . Fly ash density of 2.75  $\text{g}\cdot\text{cm}^{-3}$  was obtained with a Micromeritics 1330 gas pycnometer.

X-fluorescence measurements were made with a Philips SEM with energy dispersive analysis for major elements (calculated in oxide form) and ICP (Inductively Coupled Plasma) for minor elements. The weight percent of species found is :  $\text{Al}_2\text{O}_3$  (9 %),  $\text{CaO}$  (25 %),  $\text{Fe}_2\text{O}_3$  (8 %),  $\text{SiO}_2$  (18 %),  $\text{SO}_4$  (20 %), Cr (1126 mg/kg), Cu (2552 mg/kg), Pb (12500 mg/kg) and Zn (28500 mg/kg).

The structure and the composition (Ca/P molar ratio) of the dried powders was identified and quantified by X-ray powder diffractometry (Siemens D5000). XRD measurements were carried out with  $\text{CuK}\alpha$  radiation. The phases present were determined by comparing the patterns with JCPDS standards.

The specific area of the ground powders were determined by nitrogen adsorption using a BET method (MICROMERETICS Gemini Vacprep 061).

The shrinkage of powder was followed by dilatometry (SETARAM Setsys16/20) with 5 g constant load on the sample. In parallel, calcination experiments were carried out on a furnace of two liter volume equipped with mass loss measurement.

Figure 1: Evolution of the specific surface area function of temperature.

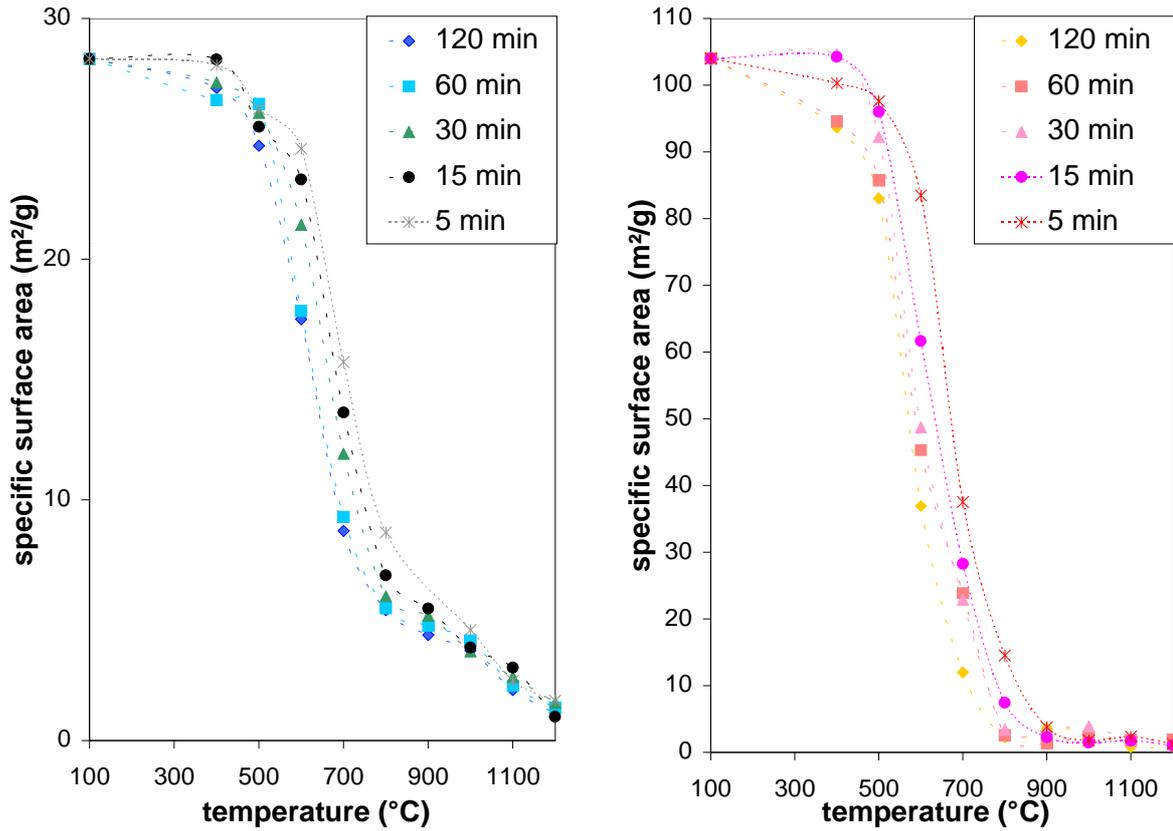
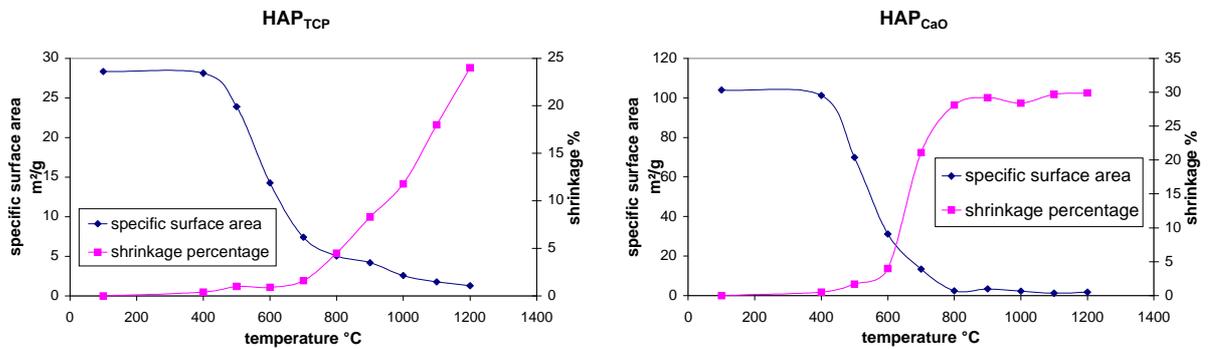


Figure 2: Evolution of shrinkage % and specific surface area as temperature function.



We have shown that calcination of the chemically treated waste gives solid residues containing apatite-like insoluble metal phosphates and help to significantly modified properties of final residues like specific surface area and density [5]. Such transformed material with stabilized trace metal ions may be considered for beneficial reuse.

Modern society is willing to pay to get rid of household refuse. Collecting, sorting and landfilling has a low cost but does take care of the immediate environmental impact of municipal solid waste. Incineration has an increased cost but contributes further to reduction in volumes and also allows for some generation of heat or energy. Further treatment of bottom ash, fly ash or air pollution control waste means another additional cost. The return is the possibility of recycling completely the initial waste. Eliminating disposal sites and transforming hazardous materials into new products for beneficial reuse is an investment for a better future. Sustainable growth in the incineration industry will only be possible with recycling of the minerals in the fly ash in civil engineering. An example of really valorisation by this way is the road construction near Dombasle-sur-Meurthe (France), where the phosphored-calcined fly ash were used as material in the road. This example will be developed during the Congress at Geneva.

#### **4 TREATMENT OF AGRS WITH PHOSPHORIC ACID AND FUTURE DEVELOPMENT.**

##### *4.1 Characterisation of AGRs.*

The AGRs are characterised by organic materials content of about 20%; the weight loss at fire to 900°C is 24% (23% to 600°C).

The element analysis is made using atomic absorption and the results are presented in Table 2.

Chemical analysis	Cd	Cr	Cu	Fe	Ni	Pb	Zn
(mg/kg)	64	210	2800	120000	600	11200	17400

*Table 2 : Chemical analysis of AGRs.*

Otherwise, by simple acidimetry, the carbonate of calcium (CaCO<sub>3</sub>) content can be estimated to 3.1 (w)%.

Tables 3-6 detail the characterisation of the four analysed AGRs.

AGR-0	AGR-1	AGR-2	AGR-3
France	Belgium	Belgium	Italy

Table 3 : Origin of samples of analysed AGRs.

Chemical analysis (g/kg)	C	H	O	N	S	P
AGR-0	112.00	15.40	103.90	5.30	15.30	--
AGR-1	348.27	49.47	151.10	5.00	traces	80.80
AGR-2	242.63	27.97	142.80	3.10	traces	32.40
AGR-3	204.40	23.00	95.40	4.80	0.90	34.80

Chemical analysis (mg/kg)	Cd	Cr	Fe	Pb	Zn
AGR-0	45	644	161814	9185	21054
AGR-1	33	73	105640	1224	7533
AGR-2	42	42	71580	1168	2439
AGR-3	15	187	70120	1728	18719

Table 4 : Chemical analysis of AGRs, Composition in heavy metals.

	AGR-0	AGR-1	AGR-2	AGR-3
Density (g/cm <sup>3</sup> )	2.5214	1.642	1.772	2.135
For diameter < 1 mm		2.072	2.176	2.244

Table 5 : Density and Specific Surface Area of analysed AGRs.

	AGR-0
LCV (kJ/kg)	4516

Table 6 : LCV of analysed AGRs.

The results show that the concentration of heavy metals in AGRs is more significant than those of fly ash. This fact explain the reason why AGRs must be treated before re-used or landfilled.

## 4.2 Results of Phosphatation of the AGRs.

### 4.2.1 Combined mud-AGRs stabilisation.

These experiments aim to the stabilisation of mixture of port mud and Automobile Grinding Residues.

Mud is characterised by a relatively important content in calcite (25 (w)%); the content in dry material is weak (32 (w)%), this dry material containing about 6 (w)% of NaCl. The content in organic materials is near to 13 (w)%; the sulphides is relatively show (0.68% S).

Chemical analysis	Cd	Cr	Cu	Ni	Pb	Zn
(mg/kg)	11	33	37	8	160	2520

Table 7 : Chemical analysis of Mud.

The process of stabilisation (2.5 (w)%  $H_3PO_4$ , 625°C) - *solidification* gives very satisfactory results of the point of view of:

- the mechanical resistance (superior to 10 N / mm<sup>2</sup>);
- the behaviour during the leaching tests.

### 4.2.2 Combined stabilisation Mud-AGR without fragmentation of the AGRs.

The AGRs are characterised by organic materials content of about 20 (w)%; the weight loss at fire to 900°C is 24 (w)% (23 (w)% to 600°C).

Chemical analysis	Cd	Cr	Cu	Fe	Ni	Pb	Zn
(mg/kg)	64	210	2800	120000	600	11200	17400

Table 8 : Chemical analysis of AGRs.

Tests of stabilisation (without solidification) have been realised at three different temperatures: 625, 750 and 875°C, and by using a concentration of phosphoric acid of 7.5 and 10 (w)% against the raw dried product.

The phosphatation have a very important effect on the leachability of the Cr, Zn and Pb heavy metals. However, for the lead, the first leachate using French X31-210 standard are not satisfying, in fact the concentrations of this metal always being over 1 mg/l. The rise of the calcination temperature is a positive factor on results.

The solidification of the “phosphated-calcined” AGRs should products satisfying materials to the leaching test; nevertheless it would require a previous thin grinding of these products, which is expansive. This fact is at the origin of the choice to achieve tests of combined “mud-AGR” stabilisation-solidification, by limiting the ratio of AGRs so that the thin component, i.e. the mud, is clearly majority.

#### 4.2.3 Results of the combined Mud-AGR stabilisation, without fragmentation of the AGRs.

The phosphatation has been achieved separately:

- for the mud (2.5 (w)%  $H_3PO_4$  in relation to the dry);
- for the AGR (10 (w)%  $H_3PO_4$ ).

Phosphated products were:

- calcined separately, and mixed;
- or mixed and calcined.

The ponderated ratio mud (dry) / AGR has been fixed to 3 / 1.

The calcination has been achieved in the usual conditions at 625 and 875°C (start cold oven, a plateau of 2 hours at the temperature).

The effective stabilisation of heavy metals is incontestable, according to the leaching test and mechanical properties.

It appears that the practice of the phosphated products mixture before the calcination does not present any inconvenience, what is evidently simpler at the industrial scale.

#### 4.2.4 Results of stabilisation and solidification of Mud-AGRs mixtures.

The calcined products have been solidified with the cement of blast furnace then, in ponderated ratio 9 / 1 (manual mixture with the cement, without attrition).

The preparation of mortars requires an important quantity of water (between 35 and 40g of water for 100g of dry) that is not on the favour of good mechanical properties.

Effectively, for all samples, the resistance to the compression after 28 days reveals to be mediocre, and on average below  $2\text{N/mm}^2$ , limit value for a solid waste (French norms).

The observation of solidified sample sections shows:

- the very important irregularity of surfaces, due to metallic particles (Fe) that break away at the time of the cutting;
- the porosity of solidified products.

## ***CONCLUSION.***

The results obtained both with incinerator fly ash and automobile grinding residues (AGRs) show that the calcination of the chemically treated waste gives solid residues containing apatite-like insoluble metal phosphates and help significantly modified the properties of final residues like specific surface, density, hardness and leachability of pollutants.

The REVASOL which is a demonstration project developed by SOLVAY lead to re-use the stabilised in road construction. For this reason, the stabilisation of heavy metals with phosphoric acid from AGRs would represents a promising way to re-use these residues.

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