

Characterization of solid residues from the incineration of expired and unused medicines

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ARTICLE INFO

Article History :

Received : 14/07/2022

Accepted : 23/11/2022

Key Words:

Expired drug ;
Waste ;
Incineration;
Bottom ash;
Fly ash;
Characterization.

ABSTRACT/RESUME

Abstract: Expired or unused medicines are potentially toxic substances that should be managed effectively and correctly to avoid their presence and accumulation in the environment. The objective of this study is to determine bottom ash and fly ash chemical composition resulting from expired and unused drugs and maximum leaching of heavy metals (copper, iron, zinc). This paper reports a detailed and complete characterization of two samples of different type of combustion ashes as bottom and fly ashes collected from incinerators located in Si Mustapha – Boumerdes. The study focused on physicochemical, mineralogical and environmental characterization. For this purpose, several techniques such as: DRX, XRF, ICP-MS, SEM, UV-visible, titrimetric and gravimetric methods were used. The physical results indicated that the unburnt rate in these residues is less than 5% which indicates a good incineration; the particle size population varies between 0.5 μ m to 50 μ m for bottom ash and between 0.5 μ m to 8 μ m for fly ash (more homogeneous particle size distribution). With regard to leaching behavior, leachates pH of the two solid residues is basic and oscillates between 8 and 11. The concentration of sulfate ions obtained is much less than 10.000 mg / Kg and the same is true for chlorides where the concentration is less than 800 mg / Kg. XRF analysis showed that the sum of the following major elements expressed as oxides: Al₂O₃, SiO₂ and Fe₂O₃ is 83.51% for fly ash and 60.68% for bottom ash. The results of the analysis of heavy metals show that the concentration of elements leached by these residues does not exceed the so-called «inert and recoverable category», namely : pb < 0.5 ppm, Cd < 0.04 ppm and Cr < 0.5 ppm, Hg < 0.2 ppm and As < 0.5 ppm; which makes it possible to classify these residues in the category of inert and recoverable waste. Results of physicochemical analyzes and leaching tests suggest that fly ash from solid waste incineration fly ash should be classified as hazardous waste and should be properly managed.

I. Introduction

In Algeria, the generation of unused and expired medicines incineration solid residues is expected to increase significantly in the future. In fact,

production of unused and expired medicines are continuously growing in the world actually estimated in high-income countries to be greater than 40% of the overall production of medicines [1]. Thus, a correct disposal of this class of waste is

assuming increasing importance. These solid residues are of particular concern because of the concentrations of potentially hazardous heavy metals, consequently, such issues have necessitated the study of the characterization of these solid residues.

Therefore, the results of this characterization would contribute to the development of adequate waste management strategies. Incineration of medical waste converts the waste into non combustible solid residue or ash [2-5]. The two main options for disposing pharmaceuticals are : landfill and thermal destruction, the latter being the best disposal method currently available. The main advantage of incineration over all other methods is the volume reduction. Incinerators reduce the solid mass of the original waste by 80% - 85% [6-10]

Expired or unused medicines are potentially toxic substances that should be managed effectively to avoid their presence in the environment. .

Municipal solid waste incineration (MSWI) produces two main types of combustion ash : bottom ash (BA) and fly ash (FA) [11,12] as our residues (expired and unused medicines) accounting for nearly 35% of the input waste mass.

Incineration is a technique adopted for 30 years in industrialized countries and it remains the most used today with more advanced techniques and very strict discharge standards. This technique allows by combustion a reduction of 70% of the mass of incoming waste and of 90% of their volume [13]; nevertheless it generates a large quantity of toxic by-products: gaseous effluents, liquid residues and solid residues [14]

This paper reports a detailed and complete characterization of two samples of different type of combustion ashes as bottom ash and fly ash collected from incinerators located in Si-Mustapha (Boumerdes). The study focused on physicochemical, mineralogical and leaching behavior characterization.

Actually, in Algeria, these solid residues are stored in industrial sites or evacuated to technical landfills (TLF) and they have never been the subject of any recovery [14]. For this, we were interested in our work in an in-depth characterization study of

bottom ash and fly ash resulting from the incineration of expired and unused drugs. The objective was to verify their behavior in the

II.2 Methods

II.2.1 Incineration protocol

- Unpacking of pharmaceutical waste (original cardboard, bottle, blisters and caps) is carried out in

environment, in order to meet the criteria in accordance with a hazardous or inert waste to be able to classify them in class I or II according to the danger they represent or in the best case to find them a potential of use as material in the production of products with high added value.

Awareness of the harmful residues from the incineration of municipal solid waste (MSW) has increased in recent years and many researchers have discussed the characteristic and reuse treatments for this residue, including fly ash and bottom ash [15-17].

II. Material and methods

II.1 Material : Description of sampling sites and incineration plant

Samples of ashes collected came from an incineration plant of industrial waste located in the city of Boumerdes (center region of Northern of Algeria). The heat treatment by incineration of three types of expired drugs used in our study (KLAVOX® tablet, KLAVOX® powder for syrup and BACTROBAN® pomade) was carried out at the Algerian company "ECFERAL" with a "Nar 5000" type incinerator equipped with a wet fume treatment system with neutralization of acid gases.(Fig.1). The solid residues generated by this process have been characterized in detail in order to verify whether they could be considered as inert or non-hazardous and recoverable waste. This complete characterization of was carried out with the aim to suggest suitable reuse strategies of this waste.

The characterization of solid residues "bottom ash and fly ash" was carried out using several physico-chemical analysis techniques such as: XRD (type PAN analytical X'pert Pro), Laser diffraction particle sizer (type Mastersizer2000), FX (brand AXIOS Pan analytical), ICP-MS (series 7700X Agilent-Technologies), SEM (type JEOL JSM-6360), UV-visible (brand Jasco), and titrimetric and gravimetric methods as analytical analysis.



Figure 1. Schematic of the typical incinerator "Nar 5000" from ECFERAL company

accordance with the internal unpacking procedure of the ECFERAL company. - The waste is weighed and introduced manually into the main combustion chamber (furnace). The combustion temperature is

between 800°C and 900°C. The fumes are treated by a wet process, they come out at a temperature of 900°C and are sprayed with clean water which will reduce the temperature and capture dust in the wet phase (fly ash) and neutralization of acid gases by soda. After neutralization, the fumes are extracted using a draft fan, then evacuated to the atmosphere through a chimney. At the end of this operation, the bottom ash is recovered directly and manually from the furnace after cooling in the open air.

II.2.2 Sampling procedure

Fly ash is a homogeneous residue, unlike bottom ash (very heterogeneous material), so it is essential to take the most representative sample possible.

The sampling was done according to Spanish standard (UNE 83-421) [18] on sampling, conservation, and transport of coal fly ashes, and according to the Standard Guide for General Planning of waste sampling (ASTM 4867-87) [19]. Before the analysis of every residue, a subsample was obtained by the quartering procedure (AFNOR) in order to minimize the risks of errors on the composition of the sample linked to its heterogeneity (Fig. 2).

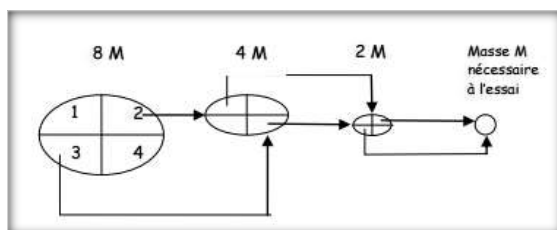


Figure 2. Schematic sampling by the quartering method

II.2.3 Characteristic study of solid residues

The properties of bottom ash and fly ash strongly depend on the nature and combustion process of the waste. It is therefore important to characterize these residues by a detailed study to verify their behavior in the environment; therefore, we will present the main physical and chemical characteristics of bottom ash and fly ash, their mineralogical compositions as well as their behavior in the environment by a leaching study.

II.2.3.1 Physical characterization

II.2.3.1.1 Aspect and color

Raw bottom ash is a porous material in the form of a heterogeneous dark gray mixture of slag. It consists mainly of the heavy and incombustible part of the incinerated waste; it is therefore an essentially mineral matrix [20]. Dry fly ash is in the form of a fine powder, similar to a cement powder

[21]. This parameter is determined by visual characterization.

II.2.3.1.2 Moisture content

This measurement makes it possible to determine the dry mass of the samples used [22].

The moisture content of bottom ash and fly ash is determined in accordance with the french standard X 31-210. Two samples with a mass of 10g of bottom ash and fly ash are placed in an oven set at 105°C until constant weight.

II.2.3.1.3 Unburnt rate

The rate of unburned solid residues was determined in accordance with the circular of 09/05/1994 relating to the elimination of bottom ash from incineration [23]. In two porcelain crucibles, two samples of mass $m = 10\text{g}$ of bottom ash and fly ash previously dried at 105°C, are placed in a muffle furnace at 500°C for 4 hours. The operation is repeated until a constant mass is obtained.

II.2.3.1.4 Particle size distribution

The particle size distribution is an important parameter in order to evaluate the capacity of solid residues to be compacted [24]. The bottom ash and fly ash powders were analyzed using a Laser dispersion particle size analyzer equipped with a dry process module which allows the measurement of particles between 200 nm and 2 mm. The operating conditions are as follows: pressure: 3.5 bars, refractive index: 1.520.

II.2.3.1.5 Morphological analysis by Scanning Electron Microscopy (SEM)

SEM observation of each bottom ash powder and fly ash requires prior preparation. After drying, each powder is deposited on a cylindrical support where the flat surface is covered with a conductive adhesive tape allowing both to fix the quantity of powder used and to ensure the conduction of the whole in contact with the electron beam. The various supports are attached to a sample port and placed on the mobile microscope stage.

II.2.3.1.6 Absolute and apparent density

The transport of raw materials represents a significant cost and the choice of one material rather than another is made in particular from the density [25]. The absolute density « ρ_s » and the apparent density « ρ_{app} » of bottom ash and fly ash are determined in accordance with Standard: NF EN 196-1.

II.2.3.2 Chemical characterization

The chemical characteristics of the solid incineration residues are used as a basis for their classification as hazardous waste or not [26].

II.2.3.2.1 Determination of pH

The pH is determined in accordance with the experimental standard AFNOR X 31-210 .

II.2.3.2.2 Determination of chlorides and sulphates

According to the circular of 9 May 1994 [23] , solid residues from incineration can be recovered in the field of civil engineering if their polluting potential is zero or low. Otherwise, they must be either treated in a maturation center to make them recoverable or eliminated in a storage center. It is accepted that chlorides and sulphates are salts, known in civil engineering as long-term destabilizing elements, and their dosage is considered important before any recovery [27]. The sulphates contained in these solid residues can react with portlandite “Ca(OH)₂” to give hydrated calcium sulphates (gypsum) which, by reaction with certain alumina phases such as non-hydrated tricalcium aluminate “3CaO • Al₂O₃” can give ettringite. The presence in large quantities of this species leads to mechanical stresses in the field of civil engineering (cracking and expansion of concrete) [27]. The chlorides react with the portlandite to give calcium chloride CaCl₂, a soluble salt which increases the porosity of the concrete. Mainly the chlorides react with the aluminate phases (C3A, not having reacted with the gypsum) to form compounds called friedel salts. This also expansive salt causes cracking of the concrete. The chlorides in the bottom ash and fly ash are measured in neutral medium with a titrated solution of silver nitrate in the presence of potassium chromate. The end of the reaction is indicated by the appearance of the characteristic red color of the silver chromate [28]. The dosage of sulphates in bottom ash and fly ash is determined by a photometric method, the sulphates are precipitated in a hydrochloric medium in the form of barium sulphate. The precipitate thus obtained is stabilized using a solution of Tween 80 (surfactant, non-ionic emulsifier), the homogeneous suspensions are measured by UV-visible spectrophotometer [28]

II.2.3.2.3 Chemical composition of major elements by X-ray fluorescence spectrometry (XRF)

- Physicochemical characterization.

According to the American standard ASTM C618 on cements, a material with the characteristic of a pozzolan if its chemical composition checks the following relation: Al₂O₃ + SiO₂ + Fe₂O₃ > 70%. The ASTM C125-07 standard, defines these materials as being siliceous or silico-aluminous materials which in themselves do not have binding

properties but which, in finely divided form and in the presence of humidity, react chemically with hydroxide of calcium at room temperature to form compounds having binding properties. With this characteristic, they can be used as an additive in the production of Portland cement. The analysis of the major elements in our residues was carried out by XRF according to ISO 29581-2, 1st edition (03/01/2010). Weigh 10 g of each sample and introduce under grinding a mixture of boric acid and microcrystalline cellulose in order to increase the cohesion of the grains of the samples. The analysis is carried out under helium to prevent the powder from flying under the effect of pumping. Pelletize with the necessary pressure for 10 seconds and pass them directly to the Spectrometer.

II.2.3.3 Mineralogical characterization by X ray diffraction

The method involves subjecting the powdered sample to a monochromatic X-ray beam and collecting the emitted diffraction spectrum . Dry the bottom ash and fly ash samples for 01 hour in an oven at 50°C, grind and sieve the samples after drying. One pellet of each sample was prepared and placed on the equipment tray for X-ray bombardment.

II.2.3.4 Environmental behavior: analysis of heavy metals by ICP-MS

Heavy metals in solid residues can leach and contaminate soil surfaces and groundwater; they are in the form of highly variable compounds. For example, lead can exist in the form of lead metal (Pb), oxide (PbO), various silicates (Pb₂SiO₄...), chloride (PbCl₂), phosphate, various alloys, and the same for other heavy metals. In environmental sciences, the heavy metals associated with the concepts of pollution and toxicity are generally: (As), (Cd), (Cr), (Cu), (Hg), (Mn), (Ni), (Pb) , (Sn) and (Zn) [14].

The analysis of heavy metals in the leachates of bottom ash and fly ash is important for the protection of the environment and for a possible recovery of the solid residues studied. The leaching test is carried out in acidulated water, with an L / S ratio = 10 l / Kg. In two beakers, 10 g of powder from each sample (bottom and fly ash) were introduced, then, 100 ml of acidulated water (pH=3) were added using hydrochloric acid to dissolve all of the heavy metals that may be present in these residues. The mixtures are subjected to stirring for 24 hours and then, are decanted and filtered. The leachates obtained (filtrates) are directly analyzed by ICP-MS .

III. Results and discussion

III.1 Results of physical analyzes

The bottom ash obtained after incineration of the expired drugs are in the form of dark gray, friable and heterogeneous granules of varying sizes (Fig 3). The fly ash obtained is a fine powder, dark black in color and soft to the touch (Fig 4).



Figure 3. Raw sample of bottom ash obtained after incineration



Figure 4. Raw sample of fly ash obtained after incineration

- The Humidity rate and the unburnt rate obtained in the two solid residues are summarized in table 1.

Table 1. Humidity rate and unburnt rate from Bottom ash and fly ash

	Bottom ash	fly ash
Humidity rate (%)	2.50%	9.1%
Unburnt rate (%)	2.64%	1.05%

According to the results recorded, we notice that the moisture content of bottom ash is very low compared to that of fly ash. These results are

predictable because the recovery of fly ash is done by wet method after incineration while the bottom ash is recovered from the furnace after cooling in the open air. They are therefore acceptable for possible recovery (<10%), however their drying is inevitable before any use. The unburnt rate obtained for bottom ash and fly ash (less than 5%) denotes a good incineration. which allows us to classify these residues in the recoverable waste category «V» according to the circular of May 9, 1994 relating to the elimination of bottom ash [20].

The particle size distribution curves of the bottom ash and fly ash before crushing and sieving (figures 5 and 6) show a wide particle size distribution of the two solid residues ranging from 1 to 1000 µm. For bottom ash, there are three granulometric populations, the largest has a diameter of around 300 µm. For fly ash there are two particle size populations, the largest has a diameter around 250 µm. After grinding and sieving, we notice that the particle size distribution of fly ash is more homogeneous than that of bottom ash (from 0.5 µm to 50 µm). Indeed, fly ash is characterized initially by fine particles. In the light of these results, we find that grinding and sieving have significantly improved the fineness of these materials, which will easily allow their incorporation into a matrix for possible recovery (figures 7 and 8). Prior studies investigated the effects of grinding fly ash and fly ash fineness on the performance of concrete containing fly ash.

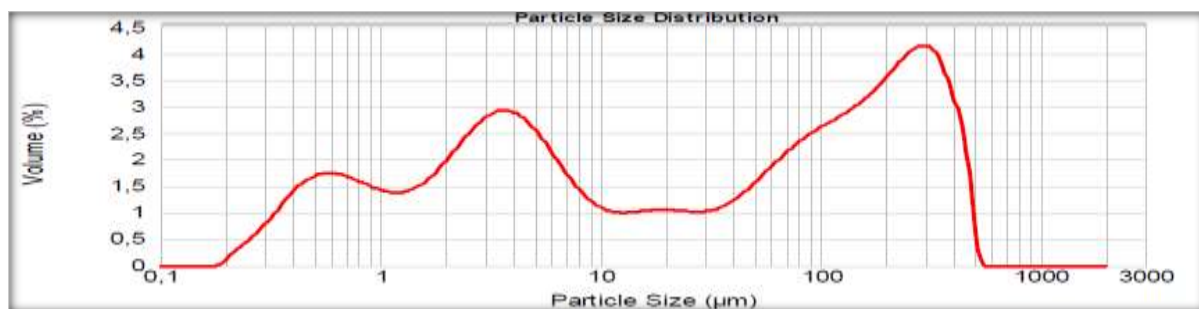


Figure 5. Particle size distribution of bottom ash before grinding and sieving

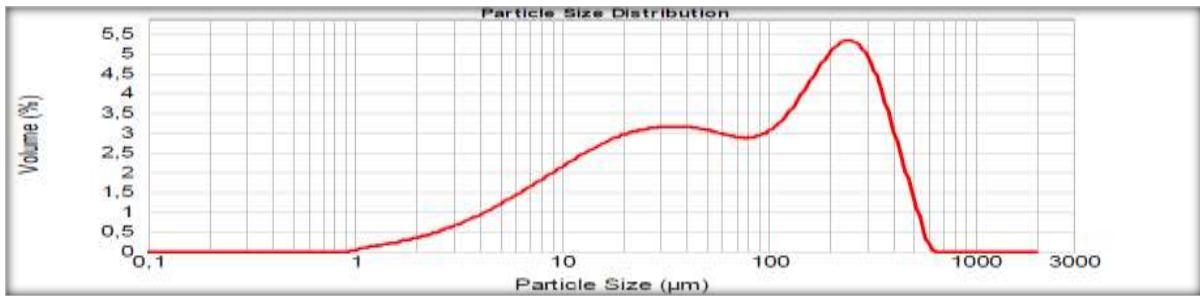


Figure 6. Particle size distribution of fly ash before grinding and sieving

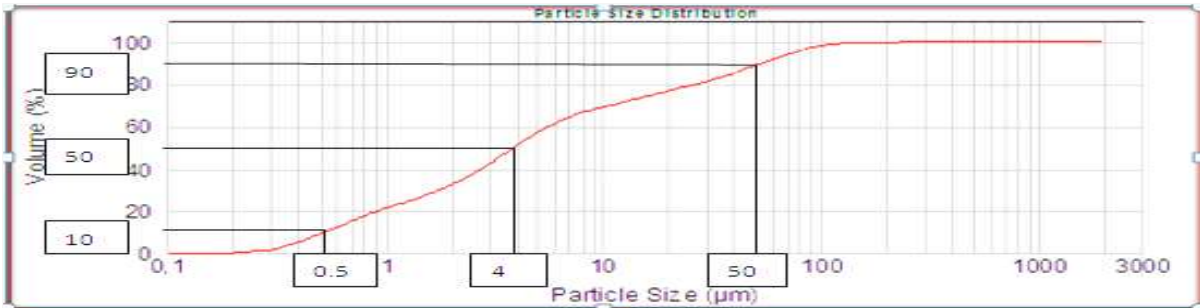


Figure 7. Particle size distribution of bottom ash after grinding and sieving at 80 µm



Figure 8. Particle size distribution of fly ash after grinding and sieving at 80 µm

- Figures 9 and 10 show the micrographs of the solid residues obtained by scanning electron microscopy (SEM) with a magnification ($\times 250$ and $\times 1000$). Bottom ash analysis shows an angular material with porous particles and fly ash analysis shows that the elements that constitute this material are in the form of quite shiny and smooth packed

balls with a wide range of fine particles of varying sizes. This compacting effect of the spherical particles of the fly ash contributes to reducing its permeability.

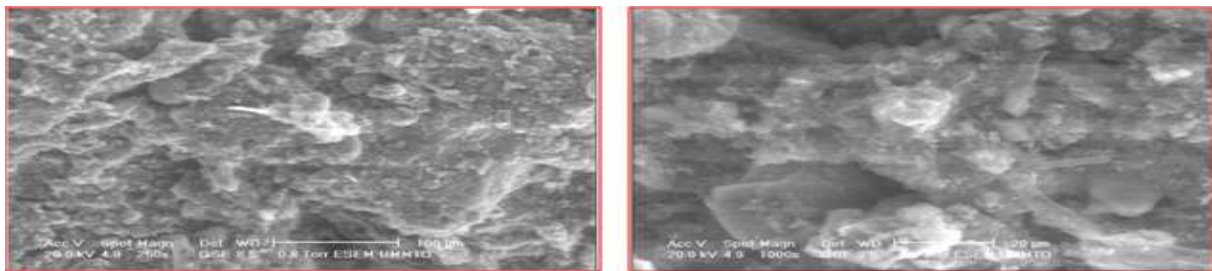


Figure 9. Scanning electron micrographs of bottom ash (magnifications $\times 250$ and $\times 1000$)

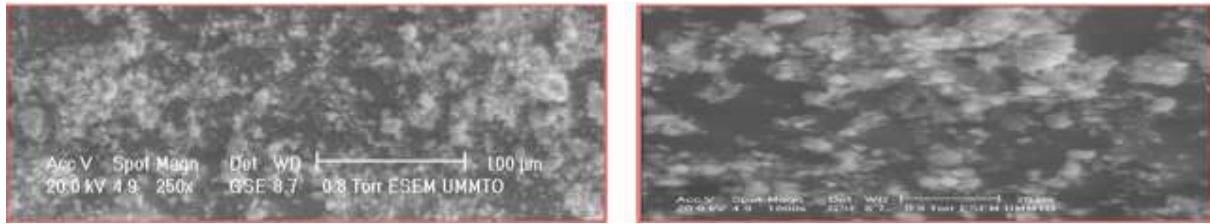


Figure 10. Scanning electron micrographs of fly ash (magnifications $\times 250$ and $\times 1000$)

- The apparent and absolute densities of bottom ash and fly ash are summarized in table 2.

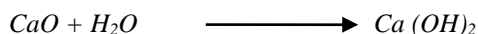
Table 2. absolute and apparent densities of Bottom ash and fly ash

	Bottom ash	fly ash
Absolute density (g/cm ³)	3.03	2.15
Apparent density (g/cm ³)	0.70	0.30

The absolute and apparent densities of fly ash are lower than those of bottom ash which makes it a rather light material; these results are directly linked to the morphology and the size of the grains of these particles which are in the form of spherical and homogeneous particles. Contrary to bottom ash, which is an angular, porous material.

III.2 Chemical analysis results

- The recorded pH results (Table 3) show that after 4 hours of stirring in water, the pH of the bottom ash and fly ash leachate is basic and fluctuates between 8 and 11. This alkalinity is due essentially to the presence of portlandite [Ca(OH)₂]; product of the hydration of quicklime "CaO" according to the following chemical reaction:



The high content of calcium oxide "CaO" found in high quantities in bottom ash compared to fly ash (Table 4) can therefore explain the high pH value of bottom ash compared to that of fly ash.

- The concentration of sulphate ions obtained for bottom ash and fly ash is well below 10,000 mg / Kg (maximum admissible concentration of "SO₄⁻" according to the circular of 9/05/1994 relating to the elimination of incineration bottom ash. The same observation for the chlorides ions where the concentration is less than 800 mg/Kg (maximum admissible concentration of «Cl⁻» for inert waste according to the European discharge directive 1999/31 / EC of the council of April 26, 1999.

Consequently, these solid residues comply with the standards adopted.

Table 3. pH, concentration of sulphates and chlorides results for bottom ash and fly ash

	Bottom ash	fly ash
pH	10.60	8.50
[Cl ⁻] (mg/Kg)	550.5	340.8
[SO ₄ ²⁻](mg/Kg)	762.5	281.25

It is important to note that the presence of high sulphate concentrations can cause ettringite formation as an hydration product of cement [23].

- The analysis results of the major elements present in bottom ash and fly ash, expressed in the form of oxides and as a percentage relative to their total content (Table 4), show that the sum of the following elements: Al₂O₃, SiO₂ and Fe₂O₃ is 83.51% for fly ash and 60.68% for bottom ash. In conclusion, it is noticed that fly ash has pozzolanic properties more important than bottom ash.

Table 4. Elemental composition of bottom ash and fly ash

Elements (%)	Solid residues	
	Bottom ash	Fly ash
SiO ₂	34.02	49.73
Al ₂ O ₃	19.55	18.75
Fe ₂ O ₃	7.11	15.03
CaO	25.69	4.5
MgO	2.90	1.38
MnO	< 0.05	< 0.05
K ₂ O	2.46	0.75
P ₂ O ₅	0.42	1.93
TiO ₂	2.90	4.65
SO ₃	0.85	0.65
Na ₂ O	0.74	1.39
PAF	2.64	1.05
Total	99.28	99.81

III.3 Mineralogical analysis results

The different crystalline phases, contained in solid wastes, were qualitatively identified by X-ray diffraction. The main phases detected in bottom ash and fly ash are as follows: the family of silicates such as quartz (SiO_2) in large quantities (both for fly and bottom ashes), the family of oxides such as hematite (Fe_2O_3) is found to be important in fly ash in contrary to the family of carbonates such as calcite CaCO_3 being important in bottom ash resulting from the carbonation of the lime:



The diffractometers obtained are shown in Figures 11 and 12.

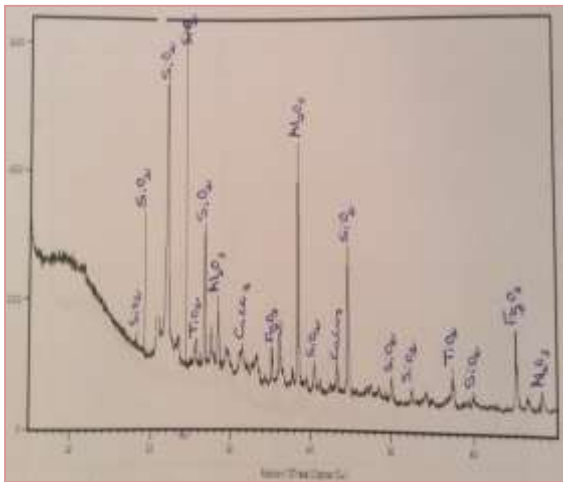


Figure 11. X-ray diffractogram of bottom ash

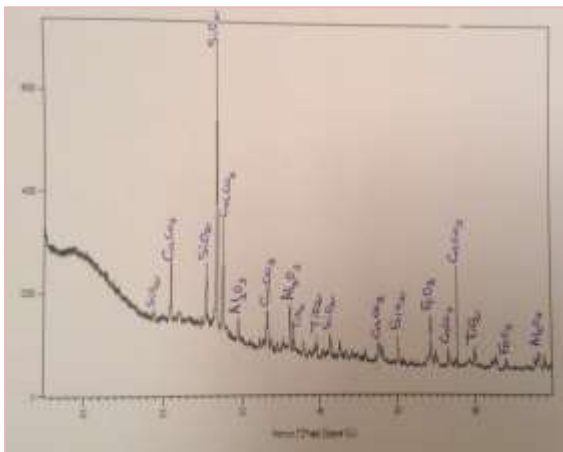


Figure 12. X-ray diffractogram of fly ash

III.4 Environmental analysis results

The elements present in the leachate from bottom and fly ash are essentially heavy metals of significant environmental interest because of the risks they involve. The obtained results are presented in Table 5.

The evaluation of the leaching behavior aims to assess the polluting potential of our solid residues, therefore in view of the results, we note that the characterization of the minority elements which potentially represent the toxic part of bottom and fly ash shows that the concentration of elements leached does not exceed the “inert waste” category of the discharge thresholds according to the European discharge directive 1999/31 / EC of the council of April 26, 1999. They are lower than the thresholds declared in the circular of 9/05/1994 relating to the elimination of bottom ash from incineration [13] for the so-called “recoverable” category such as : $\text{Pb} < 10 \text{ ppm}$, $\text{Cd} < 1 \text{ ppm}$, $\text{Cr} < 1.5 \text{ ppm}$, $\text{Hg} < 0.2 \text{ ppm}$ and $\text{As} < 2 \text{ ppm}$ (the other elements as: Ni , Zn , Fe , Cu and Co ; they are not considered in the circular). The quantity of heavy metals leached by bottom ash is less important than those leached by fly ash. From these results, we can only conclude from an environmental point of view and within the meaning of the circular relating to “bottom ash” , the solid residues studied can be considered as inert materials for later safe reuse.

Table 5. Heavy metal concentrations in bottom and fly ash leachate

Heavy metal concentration (ppm)	Solid residues			
	Bottom ash	Fly ash	Inert waste **	Recoverable waste ***
Ni (nickel)	0.0012	0.0014	0.4	/
Zn (zinc)	0.0574	0.1484	4	/
Fe (iron)	0.0020	0.0004	/	/
Cu (copper)	0.0153	0.0393	2	/
Pb (Lead)	0.0043	0.0094	0.5	<10
Cd (cadmium)	0.0002	< LD*	0.04	<1
Cr (Crome)	0.0169	0.1106	0,5	<1.5
Co (cobalt)	0.0011	0.0015	/	/
Hg (Mercury)	ND	ND	/	< 0.2
As (arsenic)	ND	ND	0.5	<2

* : LD= 0.0001 ppm (limit of detection), ND: not detected

** : European Directive standards 1999/31/CE du 26/04/1999

*** : Circular of 9/05/1994 relating to the disposal of incineration bottom ash

IV. Conclusion

The incineration of expired drugs remains a very effective means for reducing the volume of this waste and its dangerousness; nevertheless, it generates a large quantity of by-products (bottom ash and fly ash) which must be treated to reduce their impact on the environment. . The physical, chemical and mineralogical characterization as well as the leaching behavior carried out on bottom ash and fly ash made it possible to draw the following conclusions:

- The moisture content of the solid residues is low (<10%) which is acceptable for recovery, however their drying is inevitable before any use. Their unburnt rate obtained less than 5%, denotes good incineration and this allows us to classify them in the category of recoverable waste. The particle size distribution of fly ash is more homogeneous than that of bottom ash (from 0.5 μm to 50 μm). SEM analysis shows that the elements are in the form of quite shiny and smooth packed balls with a wide range of fine particles of variable dimensions. This packing effect of the spherical particles of the fly ash contributes to reducing its permeability.
- The major elements present in the residues, expressed in the form of oxides and in percentage compared to their total content, show that the sum

of the following elements: Al_2O_3 , SiO_2 and Fe_2O_3 is 83.51 % and 60.68% respectively for fly ash and bottom ash. This allowed us to conclude that fly ash has the more important pozzolanic properties.

- The main phases detected in the solid residues are as follows: the family of silicates such as quartz (SiO_2) found in large quantities in the fly ash, while the family of oxides such as hematite (Fe_2O_3) and the family of carbonates such as calcite CaCO_3 being important in bottom ash.

- The quantity of heavy metals leached by the bottom ash is less important than that leached by the fly ash. The value of the elements leached by these residues does not exceed the "inert waste" category of the discharge thresholds according to the European discharge directive 1999/31 /CE of the council of April 26, 1999 and those declared on the circular of 09/05/1994 relating to the elimination of bottom ash from incineration for the so-called "Recoverable" category.

In view of the above, we conclude that the solid residues used in our study are inert by-products to be classified in the category of recoverable waste. Consequently, considering their pozzolanic properties, their use in the field of civil engineering for the manufacture of mortars seems to be judic

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Please cite this Article as:

Bedad A., Hachemi M., Ameraoui R., Characterization of solid residues from the incineration of expired and unused medicines, *Algerian J. Env. Sc. Technology*, 8:4 (2022) 2836-2845