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Research Article

EVALUATION OF THE CAPACITY OF CEMENT KILN DUST AND FLY ASH FOR THE NEUTRALIZATION OF ACIDIC MINE TAILINGS

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ABSTRACT
The production of acid mine drainage (AMD) is considered as a serious problem for the mining industry. The oxidation of sulphide minerals causes the production of sulfuric acid resulting in a decrease in pH. This decrease causes the mobilization of metals; these very high concentrations may be a threat to the receiving environment.
Abandoned Kettara Mine (Central Jebilet, Morocco), that has been chosen as a pilot site contains 3Mt of the residues that generate acidic leachates with a pH between 2.9 and 1.5 (Nfissi *et al.*, 2011). The acidification potential (AP) of that is between 51 and 453kg $CaCO_3$/t while that the net of neutralization potential (NNP) ranges from -453 to -22.5 Kg $CaCO_3$/t (Hakkou *et al.*, 2008a).
In order to control the AMD in this mine, we opted a protocol based on the use of cement kiln dust (CKD) from the Lafarge cement of Bouskoura (Region of Casablanca) and fly ashes (FA) of the thermal power plant of Jorf Lasfar at El Jadida city (Coastal city 90km south-west from Casablanca). The main objective of this study is to enhance the value of these industrial by-products considered as wastes for rehabilitation of mine sites.
A mineralogical and physico-chemical characterization of these materials has been done and supplied promising results as to their effectiveness in stabilizing tailings. Indeed, these are highly alkaline with a high net neutralizing capacity which is in the order of 900.63 kg $CaCO_3$/t CKD and 243.75 kg $CaCO_3$/t for FA.
CKD and FA have a high neutralization potential and would constitute the main component of amendment alkali and/or covering that may reduce greatly the effects of DMA in this semi-arid climate mine and in other similar sites.

Keywords: *Kettara, AMD, CKD, FA, Characterization, Alkali Amendment, Recovery*

INTRODUCTION
Morocco knows an intense mining activity which represents 6% of gross domestic product (GDP) in 2010 (Memee, 2011), however, there are over two hundred closed sites are not rehabilitated.
During activity and after mine closure, the places of tailing storage (waste rock piles and parks tailing concentrator) can cause the production of acid mine drainage with potentially harmful consequences on the environment. This phenomenon causes the mobilization (dissolution) of heavy metals and other toxic substances that may pollute water resources and destroy natural ecosystems (Collon, 2003).
The DMA results from the oxidation of inorganic sulfur acidogens (such as pyrite, pyrrhotite, chalcopyrite...) in contact with atmospheric oxygen and water (Kleinmann *et al.*, 1981; Sracek *et al*,. 2004). Contaminated water by the DMA can come from various types of exploitation, including mines of precious metals (Au, Ag...), based metals (Cu, Ni, Zn, Pb...), coal and uranium (Plante, 2004). AMD triggers when minerals acidivores with neutralizing effects are expired.

However, in recent years, most of industrialized countries impose environmental rules more stringent during and after a mining site exploitation. A final restoration plan is required at the feasibility study of the project (e.g. Directive 019 – MSDEP, 2012).
Therefore, new treatment processes more environmentally friendly manners and new technic of effective long-term restoration are currently investigated (Bossé, 2013). Several mitigation methods can be used to prevent the production of DMA in the tailings, among which treatments are active or passive effluents (Ouakibi *et al.*, 2013), underwater deposit (Aubertin *et al.*, 2002) recoveries blankets as a capillary barrier (Dagenais *et al.*, 2003; Bossé, 2013), environmental desulphurization (Benzaazoua and Kongolo, 2003; Mermillod-Blondin *et al.*, 2005) and alkaline amendment which involves mixing generators AMD residues with alkaline materials to minimize sulphide oxidation and neutralize the acidity generated (Hakkou *et al.*, 2009b; McCullough and Lund, 2011; Mauric and Lottermoser, 2011; González *et al.*, 2012).
The Kettara Mining site located about 32 km North West of Marrakech city (31 ° 52'15 "N 8 ° 10'31" W) (Bossé, 2013) (Figure 1), which has been chosen as a pilot site for this work. This site closed since 1982 and it contains more than 3 million tonnes of tailings that were deposited over an area that about 16 hectares (Hakkou *et al.*, 2008a). The mining town of Kettara (2000 inhabitants) and social facilities are located downstream of the tailings (Photo 1).

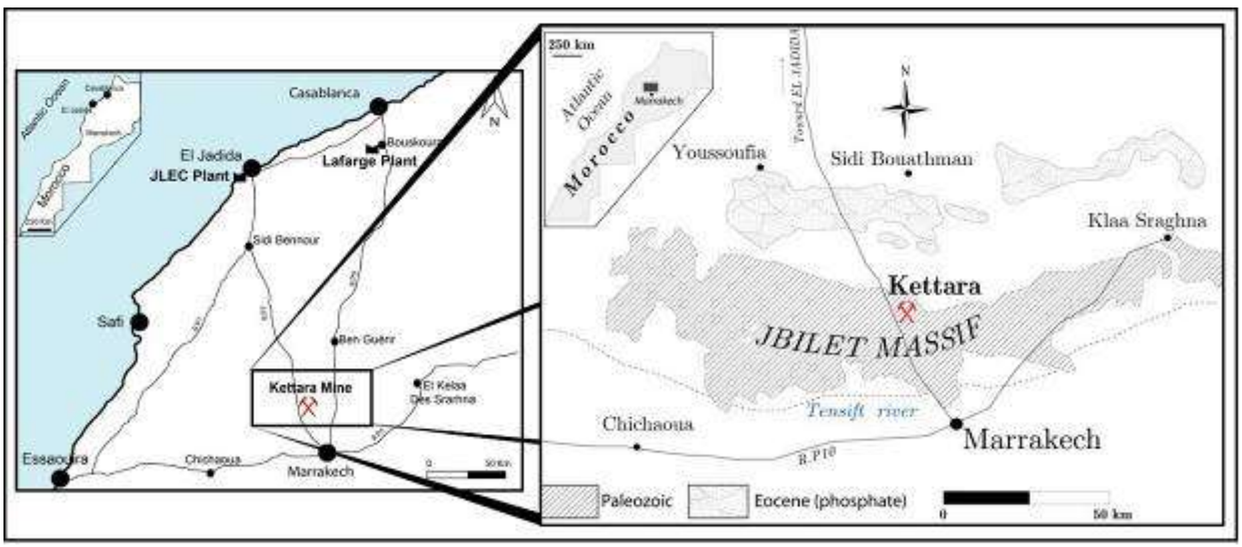

Figure 1: Location of mine Kettara, thermal Jorf Lasfar (JLEC) and Lafarge cement plant.

The Leaching tailings of Kettara produces a plunge of red brick color that is very high acidic. The pH values measured are ranged between 1.5 and 2.9 (Hakkou *et al.*, 2008a; Nfissi *et al.*, 2011). This low pH causes a high solubility which means high concentration of heavy metals. These Acid leachates are similar to those that have been produced during the test which performed in the wet cells in laboratory on Kettara's mining waste (Hakkou *et al.*, 2008b) and runoff collected in situ (Ouakibi *et al.*, 2013). The permeability tests of samples has provided a permeability between 4, 9 10^{-2} cm/s and 1.1 10^{-1} cm/s which allows to classify these tailing in the range perfectly permeable materials (Nfissi, 2013).
The results of the chemical analysis of the solid residues of Kettara reveal high levels of Pb, As, Co, Zn, Cu and Cr. The Iron, which could be associated with pyrrhotite and pyrite that present in these solid tailings with relatively large amounts (Nfissi *et al*. 2011).The Kettara thin residues are characterized by very high concentrations of SiO_2, Na_2O, K_2O, P_2O_5 compared to other residues (Nfissi, 2013).
The main sulphide minerals detected in Kettara site are pyrrhotite and pyrite, these are accompanied by small amounts of chalcopyrite, sphalerite and galena (Hakkou *et al.*, 2008a; Hakkou *et al.*, 2009a; Nfissi *et al.*, 2011).

The tailings from Kettara are highly acidity generators and thus triggers AMD which have a detrimental effect on the environment. These High concentrations of some toxic metals contaminate soil, aquatic ecosystems, wildlife and as well as human health. This mine remains an aggressive mine which generates a large AMD in the tailings park. And therefore requires control of this phenomenon by amendment and/or recovery using alkaline materials which have chosen (CKD and FA). The appropriate physico-chemical and mineralogical characterization of these materials is needs to quantify their potential neutralization of these acid generators residues.

Our papers aims are how to use industrial by-products composed of cement kiln dust (CKD) and fly ash (FA) to mitigate the impact of the AMD in the Kettara site (Central Jebilets, Morocco). The study of physical, chemical and mineralogical properties of fly ash and cement kiln dust will be made to evaluate their neutralization capacity and define the most appropriate scenarios for the control of this phenomenon in this site.

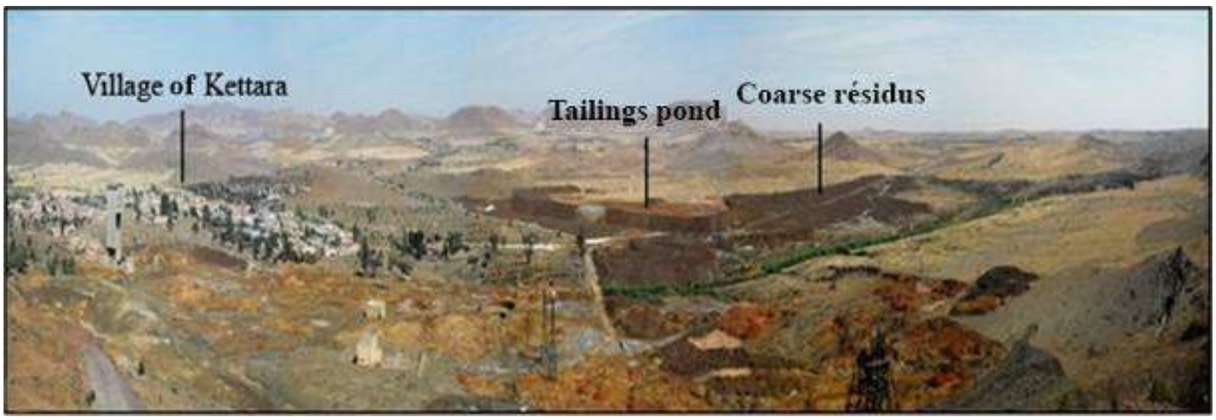

Photo 1: Situation of the town of Kettara relatively to tailings park (Lghoul *et al.*, 2012a).

MATERIALS AND METHODS

Materials Studied

Cement Kiln Dust of Lafarge (CKD)

The dust which used in this study has brought from the Lafarge cement Bouskoura near Casablanca (Figure 1). These industrial waste CKD are collected by the electrostatic filter during fabrication of the clinker which is an intermediate product resulting from combustion of the raw material of Portland cement.They have a very high potential neutralization related to the very rapid release of high concentrations of alkalis and sulfates (Doye, 2005). CKD are very rich in alkali (Na and K), chlorides and sulfates (Rhouzlane, 1997), they reduce effectively the acidity, probably due to the fineness of grain and the great reactivity of lime (CaO) they contain (Duchesne and Reardon, 1998). These conditions allow the precipitation of secondary minerals << reservoir>> such as ettringite, CSH and gypsum that can trap and stabilize heavy metals.

Fly Ash of the JLEC (FA)

Fly ash (FA) used have derived from the combustion of coal from thermal power plant Jorf Lasfar (JLEC) near the city of El Jadida (Figure 1).

These fly ash and clinker poor are stored in a quarry that equipped, according to the international standards, a few kilometers away from the power plant. When the bin becomes full, it is covered by agricultural land and vegetation cover for a good soil consolidation and integration in the landscape (El Moudni El Alam, 2005).

Analytical Methods

The pH of alkaline materials (CKD and FA) have determined by using a pH meter Type pH / Ion 510 Bench pH meter. Permeability was calculated by using Darcy's law, which connects the flow rate Q with permeability k via the hydraulic gradient, the area A and the time interval (Q = K * A * i). The total sulfur

and carbon have measured by using an analyzer ELTRA CS 800 (Carbon / Sulfur Determinator). To measure the potential acid production of these materials we adopted the standard method (Acid -Base Accounting: ABA) which developed by Smith and al., (1974) and modified by various authors (Sobek *et al.*, 1978; Lawrence and Wang, 1997; Bouzahzah, 2013).
This test measures the net neutralizing capacity which corresponds to the difference between the acid's production (AP) and the neutralization potential (NP) of samples. The AP and NP which expressed in kg $CaCO_3$/t equivalent. The neutralization potential (NP) of samples used is determined by a digestion with hydrochloric acid (1N) (Bouzahzah, 2013). The dosage of chemical elements by ICP- OES (Al, Ca, Ti, Fe, Mg, Ba, Cu, Co, Cr, Mn, Mo, Ni, Pb, Cd, Zn…) was carried out on an apparatus Perkin -Elmer Optima 3100 RL after total digestion $HNO_3/Br_2/HF/HCl$. The thermogravimetric analysis (TGA) has performed by using a Q600 SDT device TA-instruments. The quantitative mineralogy by X-ray diffraction (XRD) has been established within the Research Unit and service in Mineral Technology (URSTM) in Canada. The scanning electron microscopy (SEM) was also used to complete the information which obtained by XRD.

RESULTS AND DISCUSSION

Physico-Chemical and Mineralogical Properties of CKD

Physical Properties of CKD:

The pH values measured of the CKD leachate vary between 11.73 and 12.44. Their high alkalinity is due to the rapid dissolution of carbonates and oxides such as CaO or $Ca(OH)_2$ which generate an excellent neutralization potential (Reardon *et al.*, 1995). Work Zaki *et al.*, (2007) showed that the leachate CKD is effectived for removing copper, nickel and zinc ions, individually and/or in combination, synthetic wastewater by hydroxide precipitation.
The permeability of CKD is about $1.5.10^{-7}$ cm/s, which allows them to be classified in the category of impermeable materials (Table I). These dusts are comprised of very fine particles that are involved in the inhibition of infiltration and consequently slowing of the AMD process.

Table I: Permeability classes according to the coefficient of permeability (G.Q., 2002).

Sample	Coefficient of permeability (cm/s)
Impermeable	$\leq 6*10^{-5}$
low permeability	$> 6*10^{-5}$
	$\leq 2*10^{-4}$
Permeable	$> 2*10^{-4}$
	$\leq 4*10^{-3}$
very permeable	$> 4*10^{-3}$

Static Test (Acid-Base Accounting) of CKD:

The determination of the sulfur and carbon showed a high percentage of carbon (10.30%) relative to the sulfur (Table II). By adopting the standard test method acid-base 'ABA (Acid-Base Accounting) of Sobek *et al.*, (1978) modified the neutralization potential of CKD is 910 Kg $CaCO_3$/t and is higher than that of the waste alkaline phosphates which is in the order of 680 kg $CaCO_3$/t (Hakkou *et al.*, 2009b). The calculated acidity potential (AP) is around 9.38 Kg $CaCO_3$/t and net neutralization potential (NNP) was calculated as 900.63 Kg $CaCO_3$/t. This potential neutralization confirms their high alkalinity will neutralize the acidity of the tailings Kettara and also limit the solubility of heavy metals.

Table II: Dosage of carbon, sulfur and the neutralization potential value of CKD

	Carbon (%)	**Sulfur** (%)	**AP** (Kg $CaCO_3$/t)	**NP** (Kg $CaCO_3$/t)	**NNP** (Kg $CaCO_3$/t)
CKD	10.3	0.3	9.38	910	900.63

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Chemistry of CKD:

Analyses have performed by ICP showed a high Ca contents in the order of (31.3%) (Table III). This proves the pres ence of calcite ($CaCO_3$) and lime (CaO). Dusts effectively reduce the acidity due to the fineness of the grains and the high reactivity lime (CaO) contained in there (Mehling *et al.*, 1997), indeed the dissolution of calcite which generates an increase of pH, alkalinity and concentration of Ca (Ekolu and Azene, 2012). The calcium and other alkali present in solution reacts with hydrogen ions in the water causing of neutralization of the phenomenon of the AMD.

The CKD release high concentration of potassium sulfate and alkalinity which causes the precipitation of minerals reservoirs like ettringite, CSH and gypsum which are able to co- precipitating the metals (Duchesne and Reardon, 1998). These kiln dusts contain traces of Zn and Cr that are devoid of Co, Mo, Cd and Pb.

Table III: Chemical composition of CKD (%) by ICP

	Ca	Al	Fe	Ba	Cr	Cu	Mn	S	Mg	Zn	Ti	Ni	Sb	As
CKD	31.3	1.60	2	0.012	0.003	0.003	0.02	0.2	0.4	0.004	0.08	0.001	0.001	0.001

Thermogravimetric analysis showed that main mass loss of CKD is centered around 850 °C corresponding to the melting point of the calcite (Figure 2). This result has already been advanced by the work of Khanna (2009) which showed that the mass loss (of United States of America) is between 700 and 850 °C corresponding to the calcite.

The peaks located between 75 and 90 °C correspond to the loss of water due to the ettringite desorption or decomposition (Rhouzlane, 1997).

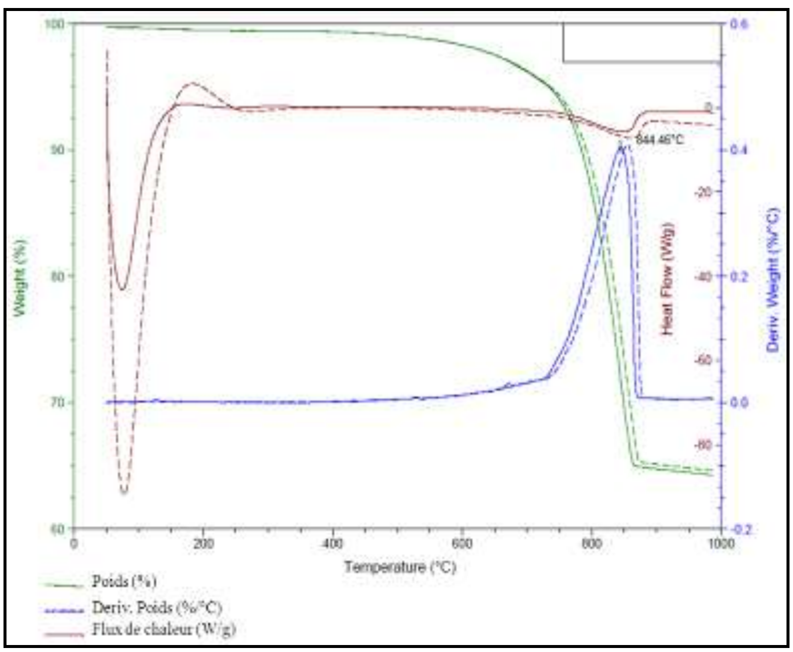

Figure 2: Thermogravimetric analysis spectra of CKD

Mineralogy of CKD:

The SEM micrographs show that the sample of CKD is composed almost entirely of calcite (88.08%) in the form of grains smaller than 10 microns (Figure 2 and Figure 3). It appears in the mapping of distribution X-ray that Calcium, Silicon and aluminum are the main components of CKD (Table IV), this is entirely consistent with the XRD results. The stoichiometry of the minerals analyzed by SEM-EDS shows that CKD also contains traces of apatite, quartz, chlorite, muscovite and corundum.

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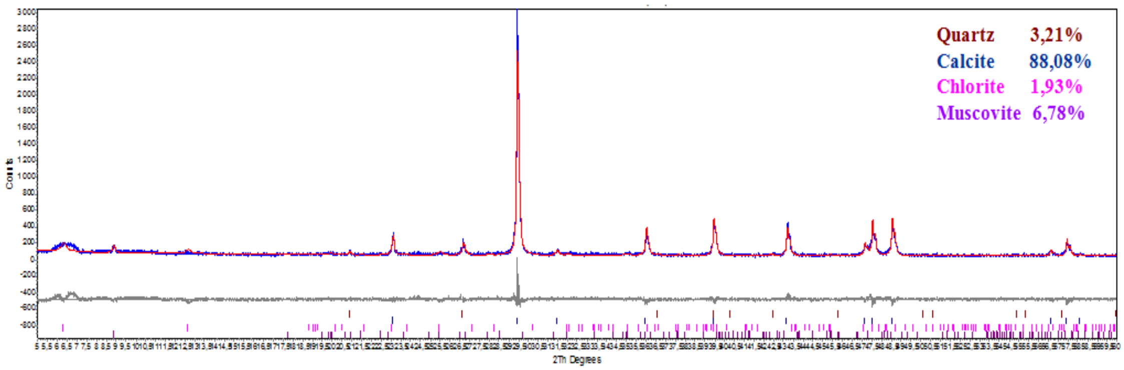

Figure 3: Mineralogy of CKD by XRD

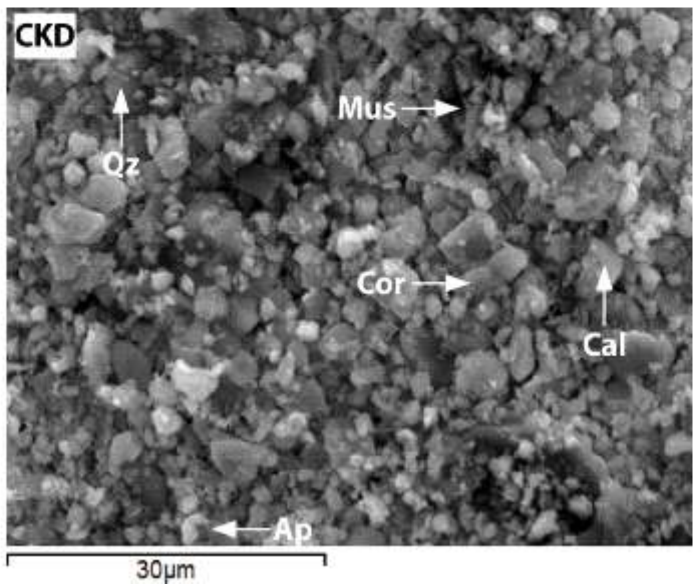

Photo 2: Imaging of CKD taken by SEM at secondary electron mode (Cal: Calcite; Qz: Quartz; Cor: Corundum, Ap: apatite; Mus: Muscovite)

Table IV: Stoichiometry of mineral CKD by SEM-EDS

	C	F	Al	Si	P	Cl	K	Ca	O	Total
Corundum (Al_2O_3)			52.93						47.07	100
Apatite ($Ca_5(PO_4)_3F$)		7.48			13.41	0.83		43.56	34.71	100
Quartz (SiO_2)				46.74					53.26	100
Muscovite ($KAl_2(Si_3Al)O_{10}(OH,F)_2$)			16.83	26.45			9.65		47.07	100
Calcite($CaCO_3$)	19.31							20.91	59.78	100

Physico- Chemical and Mineralogical Properties of FA

Physical Properties of FA:

The pH values measured in the leachates of FA is between 9.45 and 10.53. The FA may also contain substantial amounts of silicates as mullite (Ram *et al.*, 1995), which can capture hydrogen ions leading to the neutralization by forming silicates. Seoane and Leirôs (2001) have found that treatment with fly ash increases the pH gradually because of slow weathering of aluminum silicates. Mixing of fly ash with tailings reduced oxydation of pyrite and pyrrhotite because they are encapsulated by precipitation of iron as ferryhydrite on their surface (Pérez -López *et al.*, 2005). Pérez- López *et al.*, (2007) have found that the ability of fly ash to retain metals decreases at low pH leading to their release. The permeability determined in the laboratory is about $1.6.10^{-5}$ cm/s. These ashes consist of very fine grains divided in the range of waterproof materials (Table I), then they act as a barrier of infiltration that accentuate the AMD phenomenon. Their pozzolanic properties have been already demonstrated in numerous studies (Rhouzlane, 1997).

Static Test (Acid-Base Accounting) of FA:

The determination of the sulfur and carbon showed a high percentage of carbon (18.1%) relative to sulfur (Table V). The results of the acid-base test ABA '(Acid-Base Accounting) modified by Sobek and al., (1978), have shown that the neutralizing capacity of FA is in the order of 250 Kg $CaCO_3$/t. The acidity potential (AP) and net neutralization potential (NNP) calculated are respectively about 6.25 kg $CaCO_3$/t and 243.75 Kg $CaCO_3$/t. Their alkalinity is moderate compared to CKD, however it may contribute to neutralize the acidity of the tailings of Kettara.

Table V: Dosage of carbon, sulfur and the neutralization potential value of FA.

	Carbon (%)	**Sulfur** (%)	**AP** (Kg $CaCO_3$/t)	**NP** (Kg $CaCO_3$/t)	**NNP** (Kg $CaCO_3$/t)
FA	18.1	0.2	6.25	250	243.75

Chemistry of FA:

The FA are rich in Al, Fe and Ca and devoid of Cd, Sb and As, their content of Cr, Cu, Ni and Zn are negligible (Table VI). The components of the FA which essentially contribute to the pH of leachate are Ca, Mg and S, they are present on the surface of FA particles (Van der Sloot *et al.*, 1982). The solubility of many cationic elements (Cd, Cr, Cu, Ni, Pb and Zn) decreases with increasing pH (Jankowski *et al.*, 2006 in Ram and Masto, 2010).

Table VI: Chemical compositions of FA by ICP (%)

	Ca	**Al**	**Fe**	**Ba**	**Cr**	**Cu**	**Mn**	**S**	**Mg**	**Zn**	**Ti**	**Ni**	**Co**	**Mo**	**Pb**
FA	2.3	9.9	5	0.15	0.01	0.012	0.06	0.123	0.9	0.013	0.5	0.006	0.003	0.001	0.003

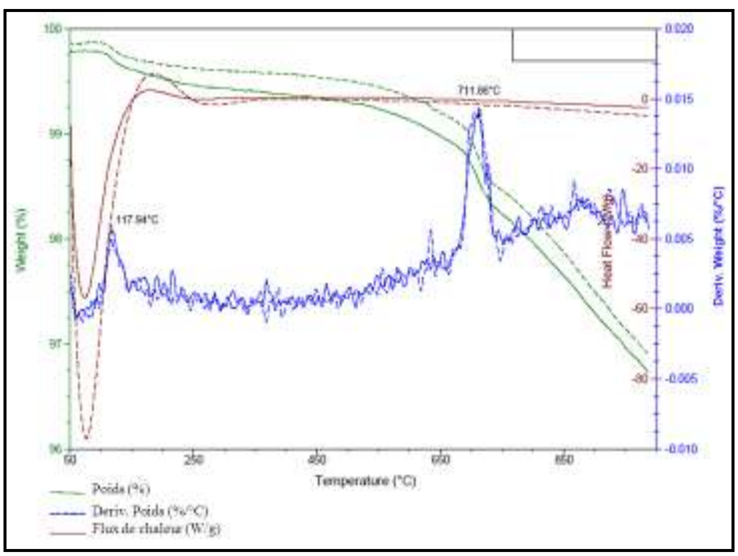

Figure 4: Thermogravimetric analysis spectra of FA

The results of the thermogravimetric analysis FA show that the first weight loss is between 100 and 200°C, which probably corresponds to a dehydration cement hydrates (CSH particular) or other hydrates (Figure 4). The second weight loss is centered at about 700°C corresponds to the decarbonation carbonates weakly crystallized. The analysis (TGA) from the work of Wrap (2007) concluded that the FA contain almost 3% of carbon at about 600-800 °C, this mass begins to decrease between 400°C to 1000°C relate to the presence of organic matter.

Mineralogy of FA:

Main phases detected by X-ray diffraction are mullite (37.95%), quartz (37.71%) and calcite (17.88%). Iron oxides are represented by the magnetite and hematite in a small amount, the gypsum is in the form of traces (Figure 5). The SEM observations also showed that they consist essentially of mullite, quartz and calcite.

The spherical mullite has a varying size, sub-micrometric to micrometric (~ 50 microns). The carbon granules have got variable shape and size (of few microns to ~ 100 microns) and often porous (Figure 3 and Table VII), the porosity is filled by mullite spherules. The hematite is also present in the form of spherules. These SEM analysis results confirm those which previously provided by XRD.

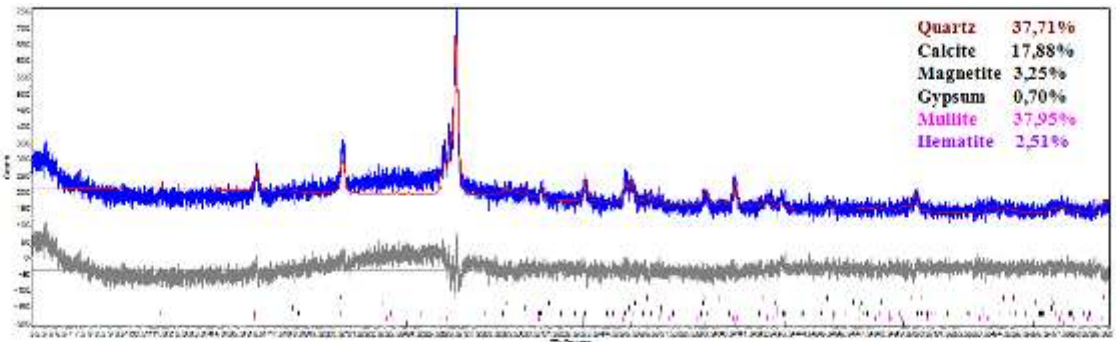

Figure 5: Mineralogy of FA by XRD

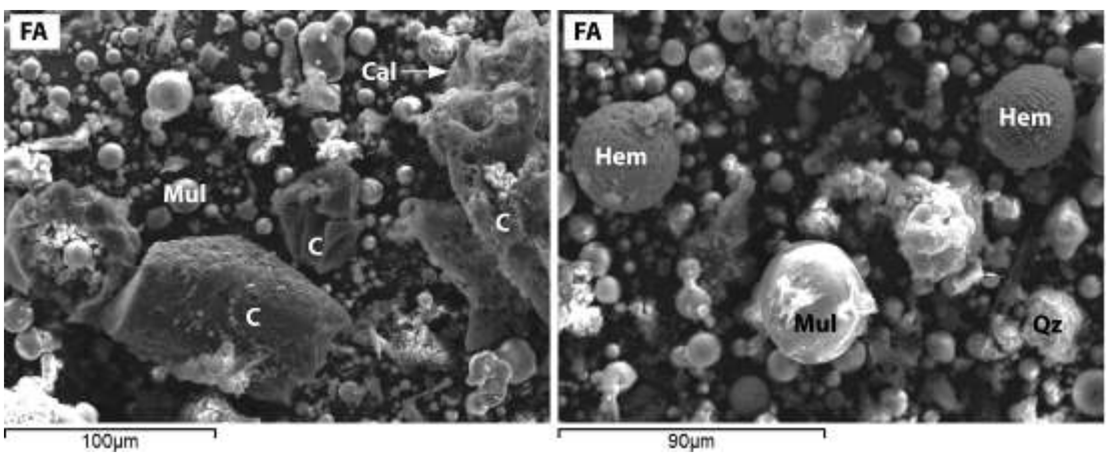

Photo 3: Imaging of FA taken by SEM at secondary electron mode (C: Coal, Mul: Mullite, Cal: calcite, Hem: hematite, Qz: Quartz)

Table VII: Stoichiometry of minerals FA analyzed by SEM-EDS

	C	Na	Mg	Al	Si	K	Ca	Ti	Fe	O	Total
Carbon (C)	100										100
Mullite ($3Al_2O_3$, $2SiO_2$)		0.63	0.56	19.89	20.43	1.27	1.88	5.4	2.93	47.01	100
Calcite ($CaCO_3$)	19.13						21.38			59.5	100
Hematite (Fe_2O_3)									77.73	22.27	100
Quartz (SiO_2)					46.74					53.26	100

Conclusion

The overall results of this study revealed that the cement kiln dust and fly ash selected produce very basic leachates.

Thus, pH values measured for the CKD leachate vary between 11.73 and 12.44, those of leachate FA are between 9.45 and 10.53. The permeability of CKD is about $1.5.10^{-7}$ cm / s while that FA is lower than it (about 1.6.10^{-5} cm/s). These materials are highly impermeable then they generate the formation of a barrier layer which will limit the infiltration of acid waters.

The acidity potential of the CKD is in the range of 9.38 kg /t; and the calculated net neutralization potential is 900.63 kg $CaCO_3$/t. The acidity potential of FA is in the order of 6.25 kg $CaCO_3$/t their neutralizing potential net (243.75 kg $CaCO_3$/t) is relatively lower than CKD. The alkalinity of FA is moderate compared with that of CKD but however they may contribute to the neutralization of the acidity of the tailings.

Recent studies have shown that CKD could be consolidated and stabilized when used in conjunction with amount of fly ash and/or other various materials (Ballivy *et al.*, 1992).

Experimental work in the field (Adaska and Taubert, 2008) and/or laboratory (Nehdi and Tariq, 2008) showed that the effectiveness of CKD increases in combination with other cementitious materials such as fly ash, which have pozzolanic or hydraulic properties that enhance effect of binder (Rhouzlane, 1997).

So CKD and FA are suitable materials, their mixture can be used as a cover layer and/or amendment in order to the stabilization of acids tailings.

At the end of these results, a new study will highlight the impact of these industrial by-products mixed and arranged in coverage and/or amendment to the attenuation of DMA through a series of kinetic tests in the laboratory. A quantitative and qualitative study will identify the most suitable protocol for the stabilization of Kettara parc mine waste tailings.

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