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Instrumental Characterization of Flyash Added Ceramic Tiles by XRF, XRD and FTIR

KEYWORDS: flyash; XRF; XRD; FTIR; building materials

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ABSTRACT *In this study experimental techniques like XRF, XRD and FTIR were used to characterize some physical and micro structural behaviors of mass and its composite containing different weight fractions of fly ash particles. XRF results show that the oxides of alumina and silica are present in major quantity and are confirmed by XRD. FTIR studies show the presence of quartz, alumina, kaolinite, hematite, and different mineral matters.*

1. Introduction
The scantiness of the ceramic ores reserves, in addition to the distance of their use place has made a strong influence on the final products costs. Besides that, high efforts in research have been made for studying new materials that are able to replace the traditional fluxing agents without changing the process or quality of the final products [1, 2]. For this reasons, several countries have interest to reformulate the body mix composition, by partial or total replacement of one of the natural raw materials. The use of waste material is considered viable only if the industrial process essentially remains unchanged and the quality and properties of the product do not decrease [1, 3-6].

The increased use of coal as a prime source of energy cannot be undermined, especially in countries like India which have sufficient coal reserves. In India almost 70% of electricity production is dependent on coal which produces a huge quantitative of fly ash as residue which is alleged as a waste product in thermal power stations. Physically fly ash occurs as very fine particles, having an average diameter of ‹10 mm, low to medium bulk density, high surface area and very high in texture.

Chemically, the composition of ash varies depending on the quality of coal used and the operating conditions of the thermal power stations. Approximately, on an average 95% to 99% of fly ash of the ash is composed of trace elements. Fly ash consists of particularly all the elements present in the soil expect organic and nitrogen [7]. The addition of fly ash into clay has the potential to reduce the cost and improving physical and mechanical properties of resulting composites.

Masse is composed mainly of silica, alumina and water, frequently with appreciable quantities of iron, alkalies and alkali earths. Two structural units are involved in the atomic lattices of most clay minerals. One unit consists of closely packed oxygens and hydroxyls in which aluminum, iron and magnesium atoms are embedded in an octahedral combination so that they are equidistant from six oxygen or hydroxyls. The second unit is built of silica tetrahedrons. The silica tetrahedrons are arranged to form a hexagonal network that is repeated indefinitely to form a sheet of composition, $Si_4 O_6 (OH)_4$. A large number of researchers determined semi-quantitative clay mineral composition on the basis of area under x-ray diffraction peak duly corrected by appropriate factors accounting for variation of scattering due to variation of angle. The literature on quantitative clay mineral analysis has been reviewed from time to time [8-11]. Later on, many others [12-15] revealed the occurrence of a wide spectrum of minerals.

2. Experimental technique
The characterization of ceramic tile is carried out with a number of experimental approaches in order to investigate all the relevant features. Masse (clay material) was obtained from the Government ceramic Institute, Vriudhachalam, Cuddalore district of Tamil Nadu and the fly ash was received from Neyveli Lignite corporation (NLC), one of the power generating plants in the province of Tamil Nadu, India. These samples were prepared into different percentage addition of fly ash samples (10 to 30%) viz. S_1 to S_4.

The masse (S1) and different addition of fly ash added ceramic tiles were analyzed for its vibrational spectra with the aid of Fourier transform infrared spectroscopy using PerkinElmer 330 series model instrument in the range 400-4000 cm^{-1} as potassium bromide pellet.

XRD patterns of masse and different fly ash added ceramic tiles were obtained on a powder x-ray diffractometer model SEIFERT J50-DE BYE FLEX-2002 with CuKα radiation having a scanning speed 10° per minute. The estimated error in the lattice parameter is of the order of ± 0.01Å. The masse and fly ash were subjected to XRF to analyze the chemical composition or elements present in the samples.

3. Results and discussion
3.1XRF characterization
It was performed to know the chemical compositions of the minerals that are present in the ceramic tiles. The Chemical composition of the masse and fly ash was given in table 1.

In terms of chemical compositions the SiO_2 was the most abundant component followed by Al_2O_3. The masse also contains a reasonable amount of potassium oxide (K_2O) 3.98%. The oxides K_2O, Na_2O, Fe_2O_3, CaO and MgO are considered fluxes. They can influence the densification behavior of the ceramic building material during firing [16].

From the chemical composition the sum of Fe_2O_3; and TiO_2 ia 1.02% (masse) and 4.24% (fly ash). Many studies have described the influence of these mineralizes in enhancing the process of sintering of ceramic matrix and the formation of mullite [17, 18] Ti^{4+} and Fe^{3+} play an important role by either substituting Al^{3+} or by their integration into the structural interstices of matrix.

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Table 1.Chemical composition of the raw materials (Masse and Fly ash).

Element Composition	Masse Concentration %	Fly ash Concentration %
SiO_2	67.31	63.02
Al_2O_3	25.57	23.47
CaO	0.456	5.513
Fe_2O_3	0.550	2.540
K_2O	3.985	0.055
MgO	0.155	1.250
MnO	0.009	0.017
Na_2O	1.300	0.425
TiO_2	0.477	1.705
CuO	0.005	0.011
SrO	0.009	0.020
Cr_2O_3	0.010	0.023
Ga_2O_3	0.003	-
Rb_2O	0.010	-
SO_3	0.127	1.760
ZnO	0.003	0.042
ZrO_2	0.017	0.104
CoO	-	0.010
V_2O_5	-	0.026
NiO	-	0.016

3.2 XRD Characterization

XRD is used to determine the mineralogical composition of the raw material components as well as qualitative and quantitative phase analysis of multiphase mixtures. The occurrences of minerals in clay were identified by comparing'd' values [19, 20]. The possible minerals with their'd' spacing values present in the absorbents are given in table 2.

No quantitative estimation phases in these samples have been made but their characterization of XRD patterns indicates the presence of Quartz, Kaolinite, Hematite, Mullite, Anorthite, Albite and Feldsper as the major phases. Further the occurrence of the above minerals in the aforesaid ceramic tiles was confirmed by FTIR study.

Table 2.X-ray diffraction data for fly ash and different proportions of fly ash additive ceramic tiles (AS_1, AS_2, AS_3, & AS_4)

d spacing Å

S.No.	AS	AS_1	AS_2	AS_3	AS_4	Mineral name
1.	--		4.42003	--	--	--
2.	4.26309	4.23852	4.23411	4.232682	4.23691	
3.	3.34867	3.3399	--	3.33244	3.32962	
4.	--		--	3.00316	--	--
5.	--	--	--	2.37773	2.37655	
6.	2.28415	2.27915	2.27568	2.27690	.27870	
7.	2.23895	2.23159	--	2.23541	2.23174	
8.	--	--	2.15467	--	--	
9.	2.12929	2.12539	2.12180	2.12317	2.12331	Quartz
10.	1.81914	--	1.81416	1.81507	1.81427	
11.	1.67265	1.66265	1.65779	1.66528	1.66881	
12.	--	1.57633	--	--	--	
13.	1.54245	1.54193	1.53896	1.53955	1.53934	
14.	1.38295	--	1.38075	1.38080	1.38186	
15.	1.37550	1.37021	1.37403	--	1.37124	
16.	--	7.11392	7.08498	7.10885	7.09443	
17.	--	3.56182	3.55931	3.561785	3.55810	
18.	--	2.33560	2.33586	2.33449	--	Kaolinite
19.	--	--	--	--	2.32915	
20.	--	1.48811	1.48746	1.48696	1.48608	
21.	1.45376	--	1.45078	1.45105	--	
22.	--	2.52497	2.54330	2.55375	2.55172	
23.	--	--	2.49059	--	--	
24.	--	1.82683	--	--	--	Hematite
25.	1.66060	--	--	--	--	
26.	1.28805	--	--	--	--	
27.	1.25650	--	--	--	--	Mullite
28.	1.22859	1.22902	--	--	--	
29.	--	3.18324	--	3.18103	--	Anorthite
30.	2.45929	--	2.44911	2.45251	2.45014	
31.	1.98129	1.97645	1.97622	--	1.97639	Calcite
32.	--	--	--	--	2.92286	Albite
33.	--	2.54566	--	--	--	Feldspar

3.3 FTIR Characterization

FTIR studies of these ceramic tiles help in the identification of various forms of the minerals present in the masse, fly ash and fly ash added ceramic tiles. The coupled vibrations are appreciable due to the availability of various constituents. Nevertheless observed bands (in the range 4000-500 cm1) have been tentatively assigned. In the IR studies of clay the Si-O stretching vibrations were observed at 790cm^{-1}, 693 cm, 538 cm-1 and 468 cm-1 showing the presence of quartz [21] The appearance of υ (Si-O-Si) and δ(Si-O) bands also support the presence of quartz [21] . The bands at 3696 cm-1, 3622cm-1 and 3450 cm^{-1}indicate the possibility of the hydroxyl linkage. However, a broad band at 3450 cm^{-1} and a band at 1633 cm^{-1}in the spectrum of clay suggest the possibility of water of hydration in the adsorbent. The inter layer hydrogen bonding in clay is assigned by a characteristics band at 3622 cm-1. Most of the bands such as 3696 cm-1, 3622.1 cm-1, 3450 cm-1, 1033 cm^{-1}, 914 cm-1, 790cm-1, 693 cm-1, 538 cm-1, 468 cm-1 show the presence of kaolinite [22]. The vibration observed at 914cm-1 indicates the possibility of the presence of hematite [23]. The presence of bands at 3696cm-1, 3622.1cm-1, 3450cm-1, 269cm-1, 1633cm-1, 1033cm-1, 914cm-1 and 790.1cm-1 indicate the possibility of the presence of illite [24], whereas 3622;1cm-1, 1633cm-1, 1033cm-1 are indicative of gypsum and 693cm-1 shows the possibility of the presence of calcite [23]. The corresponding values are given in table-4.

Table-4.Important IR bands of clay along with their possible assignments.

Band (cm^{-1})	Assignments
3696	Al-O-H str
3622	Al-O-H (inter-octahedral)
3450	H-O-H str

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1633	H-O-H str
1033	Si-O-Si, Si-O str
914	Al-O-H str
790	Si-O str, Si-O-Al str. (Al,Mg)-O-H Ai-O-(Mg,Al) str.
693	Si-O str, Si-O-Al str.
538	Si-O str, Si-O-Al str
468	Si-O str, Si-O-Fe str

4. Conclusions.

The analysis of XRF, XRD and FTIR show that fly ash added ceramic tiles is mainly constituted of silica and alumina in major quantities and iron, calcium, magnesium, potassium oxides and other elements are in minor quantities. X-ray diffraction study shows the presence of quartz, kaolinite, hematite, albite ,mullite and feldspar as major phases. The presences of above minerals were further confirmed by FTIR analysis.

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