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Evaluation of dried marine sludge-based ceramic tiles by using spectroscopy techniques

Viruthagiri* G, Ponnarasi K, Gandhimathi G and Rajamannan B

Department of physics, Annamalai University, Annamalai Nagar, Tamilnadu, India

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ABSTRACT

In this study, various proportions of tile making powder (masse) were replaced with marine sludge to manufacture ceramic tile specimens. This work describes the changes in the behavior of the masse (material) used in a ceramic industry due to addition of a marine sludge, produced in a ceramic tile processing industry in Cuddalore District, Tamilnadu, India. Compositional evaluation was assessed by XRD, SEM-EDX and FT-IR spectroscopy and the presence of impurities in the marine sludge induces changes on functional properties (e. g. refractoriness). However, those variations are easily predicted and can be accounted for.

Keywords: FT-IR Spectroscopy, Marine sludge, Ceramic tiles.

INTRODUCTION

Marine sludge are waste materials that derive from the harbour marine areas. Marine sludge are deposited material consisting of insoluble material (rock and soil particle) organic matter and remains of marine organism that accumulate on the seafloor. Marine sediments are called marine sludge. These materials are characterized by large variation in composition and by the presence of large amounts of water and organic matter [1, 2]. In some cases they are classified as toxic waste, especially when they derive from contaminated areas, since they may contain heavy metal ions (Zn, Pb, Cu, Cd, As, Hg, Ni,etc.) and organic pollutants. The disposal into landfill is expensive and not always compatible with the regulations of many countries. Marine sludge vary widely in composition and physical characteristic as a function of water depth, distance from land, variation in sludge source and the physical, chemical and biological characteristic of their environment.

Among construction materials, tradit clay-based materials are heterogeneous products that can accumulate different inorganic wastes or sub-products. Without modification of its production process or the final product properties [3,2]. Consequently the incorporation of industrial wastes or sub-products in brick and tile is becoming a frequent practice in the ceramic factories.

Ceramic tiles are commonly produced from a mixture of raw materials containing clay, flux and refractory filler. Each raw material within the body formation contributes differently to the final properties. A broad range of products varying in dimensions, dimensional tolerance, strength, decorative coatings and overall quality are produced by the tile industry. Since tile have high ratio of surface area to thickness, manufacturing process should be capable of achieving this shape in a highly productive manner. This type of tiles is usually obtained by wet grinding, dry pressing, fast drying and fast firing at suitable temperature

MATERIALS AND METHODS

The FT-IR spectra of the ceramic tile samples collected from government ceramic institute, virithachalam, were recorded in the mid region 4000-400cm^{-1} using Nicolet avatar 360 FT-IR spectrophotometer (Annamalainagar, India) with 1cm^{-1} resolution in its 100 scan mode using KBr pellet technique. The instrument was calibrated by using a standard polystyrene film each time, before recording the spectra of the samples.

In the present study, XRD pattern was recorded for only one powdered samples of ceramic tile at room temperature by using JEOL-JDX 8030 computer controlled X-Ray diffractometer system (Karaikudi, Tamilnadu, India) using Cu kα radiation of wavelength λ=1.5418 Å and Nal.(Tl) scintillation detector.

RESULTS AND DISCUSSION

Mineralogy of the raw material (S_0 and S_6).

The presence of kaolinite and montmorillonite indicate clay minerals in samples. Kaolinite is clay mineral crystallizing in the monoclinic system and forming the chief constituent of china clay and Kaolin.

The characteristic feature of Kaolinite is the O-H stretching band at about 3690 and 3620 cm^{-1} that can be a diagnostic, distinguishing kaolinite from other clay minerals. Such a clear observation was noticed in almost all the sample. The absorption band at around 3695 and 3620 cm^{-1} are attributed to the stretching vibrations of inner surface OHs and inner OH respectively. The absorption band at 912 cm^{-1} is assigned to bending of Al_2-OH. Shows the table 1 the samples S_0 to S_6 the silicate band is found to be centered around 1030 cm^{-1} with very strong intensity indicating red clay origin of the Kaolinite clay used in making the ceramic tiles [4].

From the figure (S_0 to S_6) none of them gives absorption around 750 cm^{-1} with an intensity comparable to that of the 779 cm^{-1} band indicating that well ordered clay minerals are absent in these samples.Montmorillonite is a very soft phyllosilicate mineral that typically forms in microscopic crystals, forming clay. Montmorillonite a member of the smectite family is 2:1 clay, meaning that it has 2 tetrahedral sheets sandwiching a central octahedral sheet.

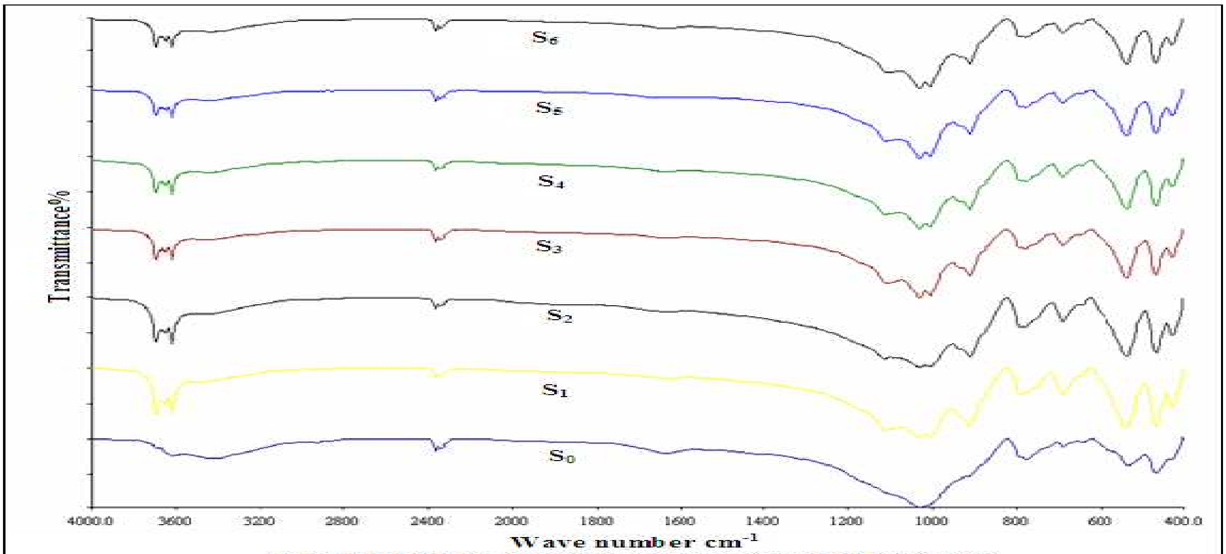

Figure 1.1 FT-IR Spectra of ceramic tile samples (S_0-S_1)

Heavy minerals

The accessory minerals, magnetite and hematite have absorption bands at 580 cm^{-1} and 540 cm^{-1} respectively. These iron oxides magnetite and hematite are of low abundance in sediment minerals and sometimes it might be difficult to identify in the infrared spectra due to the overlapping absorption bands of the silicates [5].

Quartz

Quartz is one of the commonest of all rock forming minerals and also most important constituent of the earth's crust. It is the second most abundant mineral in the earth's crust.

It occurs in crystals of the hexagonal, commonly having in the form of a six-sided prism terminating in a six-sided pyramid; the crystals are often distorted and twinge are common. Quartz is a common constituent of granite, sandstone, limestone and many other igneous, sedimentary and metamorphic rocks. All these samples (S_0 to S_6) show the absorption band of 779 and 694 cm^{-1} which are attributes to Si–O indicates of quarts [6,5,7].The bands at 468 cm^{-1} and 430 cm^{-1} have been assigned to Si-O-Si and Si-O bending mode respectively. The intensive band found around 470 cm^{-1} is due to Si-O-Si bending vibrations [8].

X-Ray Diffraction

From the diffractogram (Figures 1.2 and 1.3) the sample(S_0,S_1,S_4,S_6) have quartz, feldspar, kaolinite, anorthite, montmorillonite, calcite, orthoclase, illite and albite minerals which, were identified from the values of d- spacing 4.2592 Å, 2.1290 Å, 1.9812 Å, 3.3463 Å, 2.2379 Å, 1.7931Å, 3.7421 Å , 3.1924 Å, 3.4889 Å, 3.2468 Å, 2.4561 Å, 1.5419 Å,2.2827 Å,2.1601 Å ,1.8185 Å, 1.8502 Å and2.9343 Å respectively.

These results were obtained from the search matching of the d- spacing values in the JCPDS file (1999). [10]) have studied the organic mineral interaction in marine sediments using XRD diffractogram and identified the minerals quartz, orthoclase, smectite, calcite and albite. [11] also reported that the montmorillonite is a kind of smectite clay mineral probable available in the sediment environments.

Table 1 Infrared absorption frequencies (cm^{-1}) for marine sludge (S_0) and masse (S_1) and different proportion of ceramic tiles (S_2, S_3, S_4, S_5 and S_6)

S_0 Only sludge	S_1 Only masse	S_2 10% - 90%	S_3 20% - 80%	S_4 30% - 70%	S_5 40% - 60%	S_6 50% - 50%	Tentative assignments	Corresponding minerals
3690	3696	3696	3695	3695	3696	3695	OH stretching internal surface free (Al_2O-H)	Kaolinite
-	3676	3675	3676	3675	3675	3676	-	-
-	3669	3669	-	3669	3669	3670	Deformation internal surface free OH ant symmetric mode (Al_2O-H)	Kaolinite
-	3650	3650	3650	3650	3655	-		
3619	3620	3620	3620	3620	3620	3620	O-H stretching of inner hydroxyl group	Kaolinite
3600	-	3601	3601	-	3600	3600	-	-
-	3568	3568	3568	3566	-	-	-	-
2924	2926	2926	2926	2923	2924	2929	Organic matter	-
2852	2853	2854	-	2852	2853	-	Organic matter	-
2361	2360	2361	2360	2360	2360	2360	-	-
2343	2343	2343	2343	2342	2342	2343	-	-
2272	-	-	2273	-	-	2273	-	-
1636	1633	1632	1636	1636	1637	1636	Stretching vibration of free hydroxyl group	Kaolinite
-	1114	1115	1113	1114	1113	1112	Si-O -band	-
1032	1031	1031	1031	1031	1031	1031	Si-O stretching	Kaolinite
-	1006	1006	1006	1006	1006	1006	Si-O-Si symmetric stretching	Kaolinite
-	-	-	936	936	935	937	-	-
-	912	912	912	912	912	912	Al_2O-H deformation (internal surface OH)	Kaolinite
-	793	790	-	-	-	-	Si-O of quartz	Quartz
778	-	-	779	780	779	779	Si-O of quartz	Quartz
694	694	694	694	694	694	694	Si-O of quartz	Quartz
669	-	670	669	669	669	669	-	Quartz
647	651	649	649	649	648	648	-	Feldspar
534	536	536	537	537	537	537	Si-O–Al (or) Fe_2O_3	Hematite
466	468	467	468	468	468	468	Si-O-Si bending	Quartz
432	430	429	430	431	430	431	Si-O of mixed vibration	kaolinite

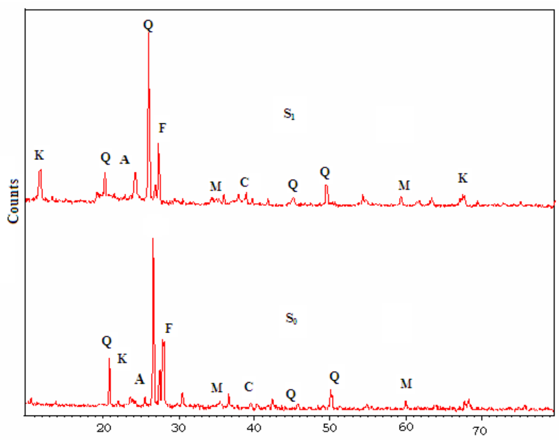

Figure 1.2 X-Ray diffraction pattern of marine sludge (S_0) and masse (S_1)

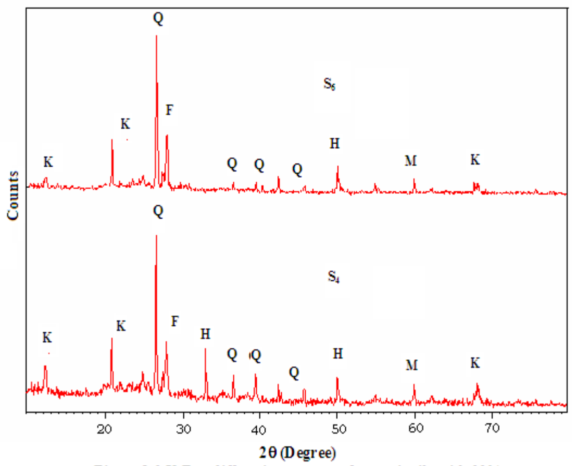

Figure 1.3 X-Ray diffraction pattern of ceramic tile with 30% of marine sludge S_4 and 50% of marine sludge S_6

The reason for the absence of the few minerals in the analysis which were identified through FT-IR is due to the disorderness (loss of crystalline nature) of the respective minerals.

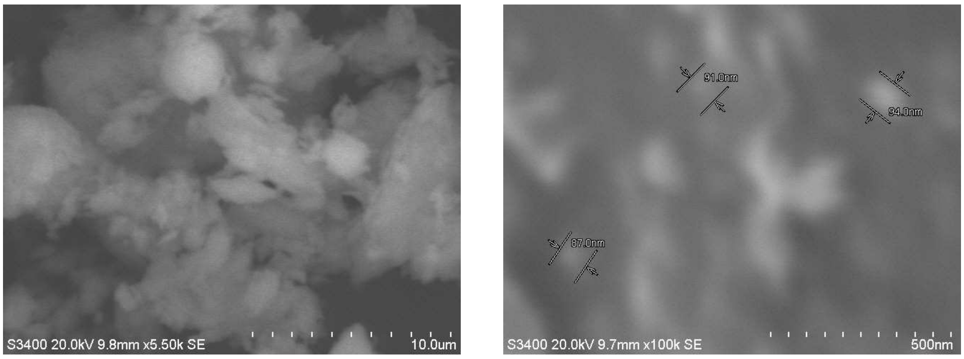

Figure 1.4 SEM pictures of sludge sample

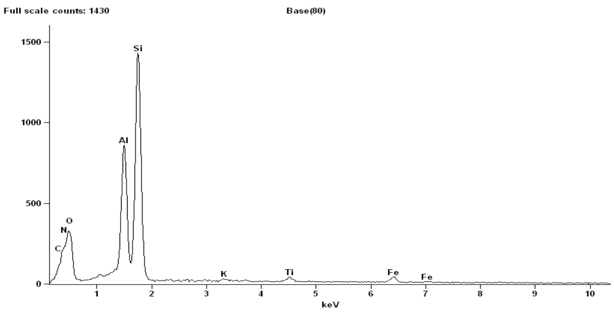

Figure 1.4a EDX spectrum of sludge sample (S_0)

At the same time, few minerals like, albite, anorthite and calcite were identified only in XRD analysis. Thus from these observations, the decreasing trend in the main reflections indicate its lowering of the crystalline nature of the respective minerals. Hence, the present results are having the same trend as observed in FT-IR analysis.

From the results reveal in the sample (S_0) (marine sludge) the clay minerals contents are very small than tile powder (masse S_1) the sample S_0 is related to the scarcity of eruptive igneous rocks in the marine area.

SEM with EDX Analysis.

The figure (1.4) shows the central mode is centered at approximately 90nm diameter for the marine sludge sample. In addition, the vast majority of particle<100 nm diameter are essentially non-porous in nature [13].

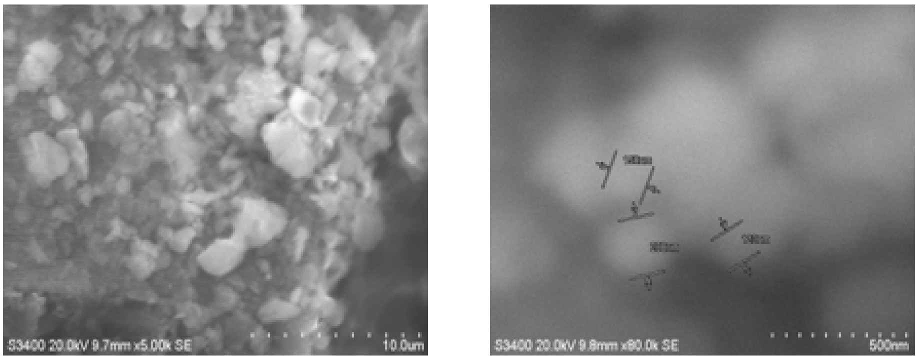

Figure 1.5 SEM pictures of masse sample

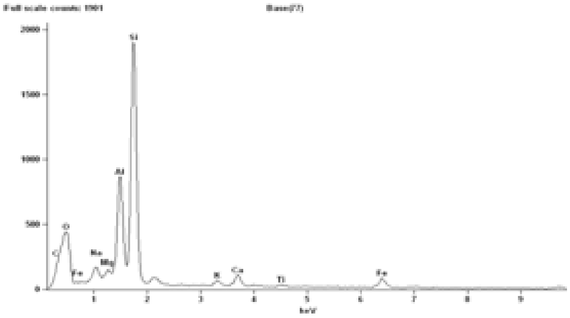

Figure 1.5a EDX spectrum of masse sample

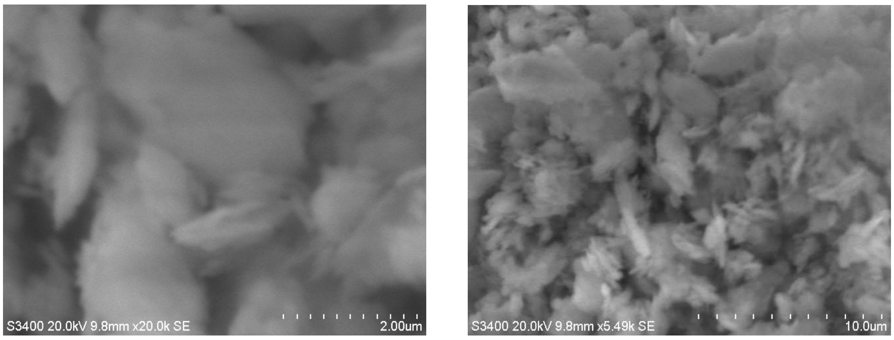

Figure 1.6 SEM pictures of ceramic tile contains 30% of sludge

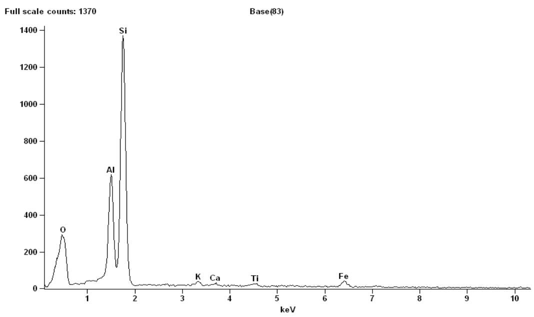

Figure 1.6a EDX spectrum ceramic tile contains 30% of sludge

The morphology structure of sample S_0, Showing isolated quartz and amorphous Fe hydroxides. The particles surfaces display the quality overgrowth. The marine sludge sample contains a lot of quartz, has a maximum in the intermediate density fractions. The sample S_1 is centered at approximately ranging from 125 nm diameter for the masse sample .In addition, the vast majority of particle (<1µm diameter) area essentially non–porous in nature. However all particles examined appeared to be primarily spherical in shape and samples S_4 and S_6 are shown in figures 1.7, 1.7a and 1.8, 1.8a showing isolated quartz with amorphous Fe hydroxide. The particle surfaces display a feathering appearance, possible reflecting the presence of quartz and iron. The observed result is in close agreement with [14]. The micrographic method is a powerful approach for isolating organic mineral aggregates. Regarding the elemental analysis (EDX analysis) confirms the presence of oxygen, silicon and aluminium in all the four samples in major proportions. Titanium and iron in trace amount.

Table 1.2 Elemental concentration of marine sludge (S_0) and masse (S_1) ceramic tiles Powder (masse) added with 30% (S_4) and 50 % (S_6) marine sludge.

ELEMENTAL CONCENTRATION	ATOMIC WEIGHT%			
	S_0	S_1	S_4	S_6
O	40.1067	50.4933	46.5325	29.26
Al	11.91	13.1067	9.0075	6.21
Si	27.2567	34.07	37.795	31.92
K	0.2067	0.62	1.3	055
S	-	0.36	-	-
Ti	0.2566	0.41	0.625	0.14
Ca	-	-	5.04	1.1975
Fe	1.2633	1.1367	2.705	6.9075
C	45.25	-	-	37.915
N	11.75	-	-	-
Mg	-	-	2.095	0.27
Nb	-	-	0.46	-
Na	-	-	-	1.22

CONCLUSION

India is one of the leading countries in ceramic tile production all around the world. Ceramic tiles containing marine sludge additives up to 50 percent were produced and characterized by using different techniques.

The various types of minerals and organic compounds were detected in the sludge by the FT-IR analysis. The morphology of the sample powders were detected during SEM examination. EDX analysis was also applied on treated marine sludge. Al, Si and C, were mainly determined. The existence of Na, Mg, K, S, Ti, Ca, Fe and Nb were less than 5% where the elemental mapping study have indicated some non uniformities to these elements in the powder structure. The production of ceramic tiles by using marine sludge additives has been successfully achieved in this work.

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