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RESEARCH ARTICLE

FT-IR TECHNIQUE IN INSULATING FIRE BRICKS STUDIES

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ABSTRACT

Fourier Transform infrared spectroscopy (FT-IR) has been carried out to analyze the both chemical and mineralogical composition of the IFB (Insulating Fire Bricks) samples. In the present study, the insulating fire bricks have been prepared by mixture of kaolin, plastic clay and sawdust materials. FT-IR technique has been used to find the mineralogical transformations of clay during firing temp in the range of 900°C - 1200°C. The FT-IR results indicate that insulating fire bricks have different mineralogical composition i.e., namely kaolin, quartz montmorillonite, hematite respectively. The increasing firing temperatures lead to the disappear of kaolinite (1114 cm^{-1}) and the development of quartz (1096 cm^{-1}). Mullite is developed from kaolin clay mineral and this is confirmed by FT-IR spectra

INTRODUCTION

Kaolinite is an economically important clay mineral that is common in the weathering, digenetic, hydrothermal, and very low grade metamorphic environments [1]. Kaolinite is one of the most important material in the ceramic industry with very different applications such as pottery, tiles, cement and bricks [2]. Over the last decade many researchers have investigated the properties of various materials have included solid waste treatment sludge, dam sediments, slag from steel production, slag from incinerator residues and various other inorganic wastes potential application include light weight aggregate in bricks, tiles and other construction products [3-5]. Sawdust is a dry product of saw mills, where it is produced from the cutting of wood. The residual wooden powder is known as Sawdust. Sawdust is a minor fuel and is used to warm houses [6]. Sawdust is a minor waste from wood based industry, with high silica content. The sawdust contains above 50% silica with highly porous light weight. Sawdust has been applied as an amendment in many materials. This is due to its high porous insulating property refractory brick manufacture of insulation, flame retardants, etc… [7-9] during the firing process, these minerals change their structure, the decompose and finally new minerals are formed. The technological properties of these products are influenced by the chemical and mineralogical composition of natural clay materials as well as firing conditions [10]. One of the most important and value added applications of the infrared spectroscopy study is the identification of the minerals in the IFB samples [11]. FT-IT technique is used to distinguish the different type of clay minerals and to derive information concerning their structure, composition and structural changes upon chemical modification [12].The mineralogical compositions has a major influence on uses of clay. The present study was carried out to analyze the chemical composition of IFB samples at various firing temperature (900°C - 1200°C) using FT-IR spectral analysis technique.

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MATETIAL AND METHODS

Preparation of samples

The raw materials (kaolin, plastic clay and sawdust) were collected from Government Ceramic Industries, Viruthachalam, cuddalore district, Tamil Nadu, India. The materials were mixed with sufficient amount of water for the formulation of ceramic pastes and kept at room temperature. The test specimens were prepared by hand shaping, molding and hand pressing. The samples obtained with these techniques were 93mm x 29mm x 29mm rectangular bars. The specimens were dried at room temperature for a day and then sun dried for 3 days. Finally the specimens were fired in an electrical furnace, at temperature ranging from 900°C - 1200°C after reaching desired temperature, the specimens were kept in furnace for 4 hour and cooling occurred by natural convection after it was turned off. The fired samples were subjected to FT-IR technique. Sample preparation of mixture of kaolin, plastic clay and sawdust materials ratio is shown in Table 1.

Table 1. The mixture of kaolin, plastic clay and sawdust materials ratio

S.No.	Kaolin (%)	Plastic Clay (%)	Sawdust (%)
		Group I samples	
S1	53	20	27
S2	46	27	27
S3	40	33	27
S4	33	40	27
		Group II samples	
S5	46	27	27
S6	50	27	23
S7	53	27	20
S8	56	27	17

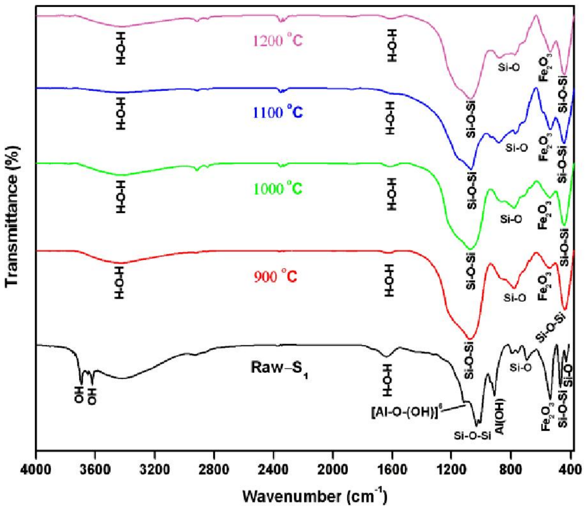
Analytical technique

FT-IR

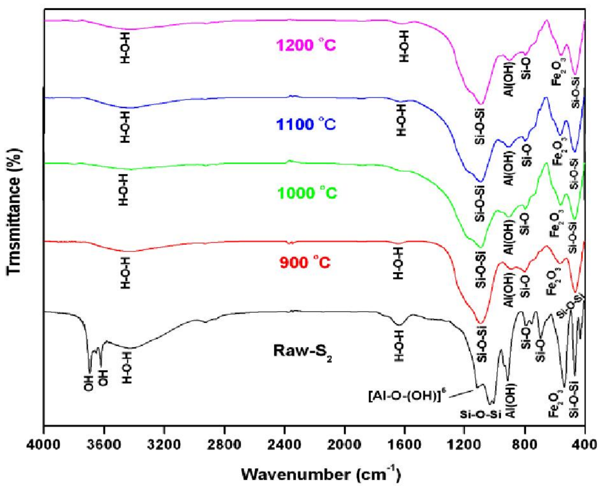
The infrared spectra of the samples were recorded using KBr pellet technique in the region 4000-400 cm^{-1}. The FT-IR measurements by using PERKIN ELMER RX1 series FT-IR spectrometer available at Department of physics, Annamalai University, Annamalai Nagar Tamil Nadu, India.

RESULTS AND DISCUSSION

The FT-IR spectra of IFB unfired and fired at different temperatures (900-1200°C) for group I (S_1-S_4) and group II samples (S_4-S_8) were presented in Figs.1 and 2.



(a)



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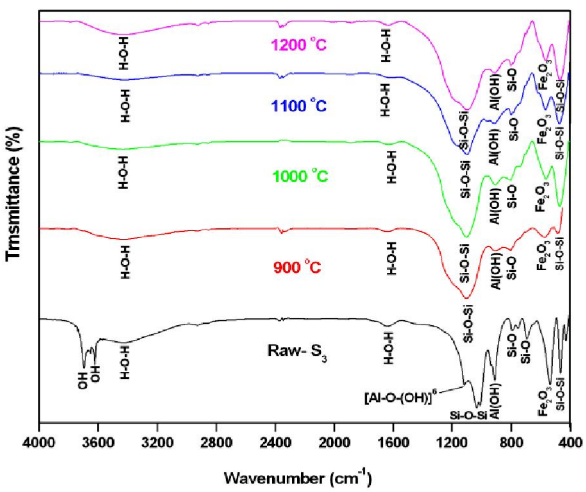

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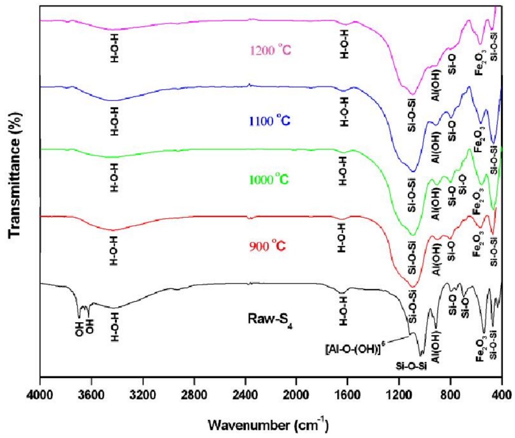

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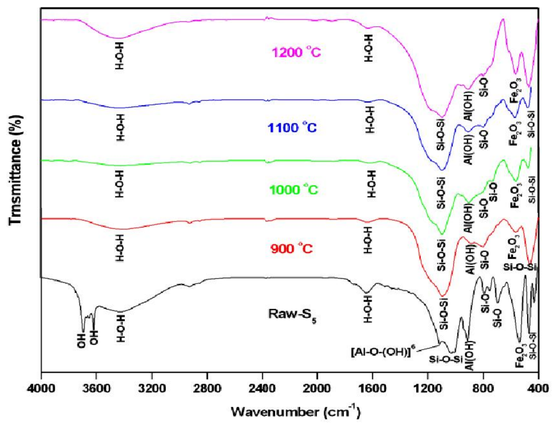
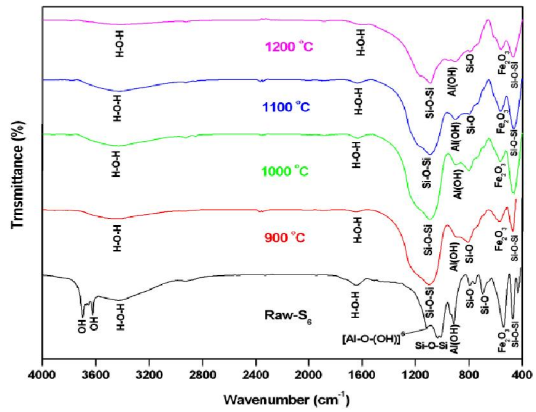
Fig. 1. The overlap of FT-IR spectra for IFB unfired and sintered at different temperatures group-I (S_1-S_4) samples



(a)



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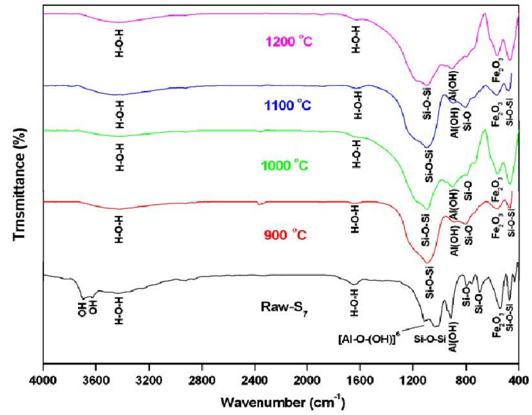

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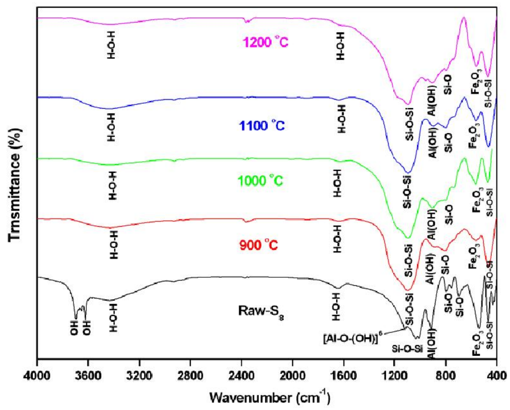

(d)

Fig. 2. The overlap of FT-IR spectra for IFB unfired and sintered at different temperatures group-II (S_5-S_8) samples

The absorption observed in the spectra with their relative intensity, minerals name and tentative assignments were presented in Tables 2 and 3. The minerals such as quartz, kaolinite, hematite, and montmorillonite for different compositions were identified. The FT-IR spectra indicate quartz and kaolinite as the major constituents and other minerals are in minor level.

The presence of quartz in the samples can be explained by Si-O-Si asymmetrical bending vibrations in the ranges 461-467 cm^{-1} [19]. The strong absorption band observed at 755 cm^{-1} and 695 cm^{-1} belongs to Si-O symmetrical bending vibration of quartz. Si-O symmetrical stretching vibrations in the range 755-795 cm^{-1}[14, 15]. The bands at 470 cm^{-1} and 431 cm^{-1} have been assigned to Si-O-Si and Si-O bending mode respectively. The intensive band found around 469 cm^{-1} is due to Si-O-Si bending vibrations [12]. Illite and kaolinite are the main clay minerals which is extremely common in soils and in natural aerosols. The strong absorption band observed at 539 cm^{-1} is due to stretching vibration of Si-O–Al (or) Fe_2O_3 in the presence of hematite [13]. The FT-IR spectral data with their corresponding tentative assignments are presented in Table 2. The overlap of FT-IR spectra of unfired and IFB materials in group I (S_1-S_4) and group II samples (S_5-S_8) are shown in Figs (a-c).

Fired at different temperatures (900-1200°C) in group I (S_1-S_4) and group II samples

FT-IR spectra of samples sintered at different temperature allows for studying the minerals transforming the breakdown of the clay structure and the formation of new materials in the high temperature region. The firing of clay raw materials to ceramic is widely documented in the ceramic industry [22]. FT-IR spectra of clays samples fired at 900, 1000, 1100 and 1200°C for 4 hour in laboratory conditions. In firing calcareous clay above 900°C, the free lime react

Table 2. FT-IR absorption frequencies (cm^{-1}) for IFB unfired samples and sintered at different temperatures

Unfired	900ºC	1000ºC	1100ºC	1200ºC	Tentative Assignments	Minerals Name
3692	-	-	-	3692	OH-Stretching	Kaolinite
3676	-	-	-	3678	OH-Stretching	Kaolinite
3670	-	-	-	-	OH-Stretching	Kaolinite
3656	-	-	-	-	OH-Stretching	Kaolinite
3650	-	-	-	3651	OH-Stretching	Kaolinite
3620	-	-	-	-	OH-stretching of inner hydroxyl group	
3421	3423	3428	-	3424	H-O-H adsorbed water	-
-	-	-	-	1708	H_2O deformation adsorbed water accompanied by rotation transitions	Kaolinite
1642	1640	1628	1630	-	δH-O-H	
1114	-	-	-		[Al-O-(OH)] 6	Montmorillonite
-	1095	1092	1092	1092	Si-O-Si	-
1032	-	-	-	-	Si-O-Si	Kaolinite
1009	-	-	-	-	Si-O-Si	Kaolinite
937	-	-	-	-	Al-(OH)	Kaolinite
912	-	902	914	917	Al-(OH)	Kaolinite
795	800	799	796	798	Si-O of Quartz	Quartz
755	-	-	-	778	Si-O	Quartz
695	695	695	695	-	Quartz	Quartz
538	-	-	543		Fe_2O_3 (or) Si-O-Al^{VI}	Hematite
469	469	470	473	477	Si-O-Si of bending	Quartz
-	-	463	461	467	Si-O-Si	Quartz
431	-	-	-	-	Si-O-of mixed vibration	-

Raw materials in group I (S_1-S_4) and group II samples

The presence of kaolinite and montmorillonite indicate clay minerals in group I and II samples. Kaolinite is the clay mineral crystallizing in the monoclinic system and forming the chief constituent of china clay. Strong bands between 3694-3620 cm^{-1} indicate the possibility of the hydroxyl linkage. However, broad absorption bands at 3432 cm^{-1} in the spectra of IFB samples suggest the possibility of water of hydration in the adsorbent. The absorption band at 912 cm^{-1} is assigned to bending of Al_2-OH. 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 IFB samples. Most of the bands such as 3694 cm^{-1}, 3677 cm^{-1}, 3669 cm^{-1}, 3650 cm^{-1}, 3620 cm^{-1}, 1032 cm^{-1}, and 912 cm^{-1}show the presence of kaolinite [12,19]. Kaolinite is the major constituent of clays which gives sharp absorption bands in the 3700–3600 cm^{-1} region. 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 is one of the non-clay mineral, which is common and invariably present in all the samples. The Si-O bonds are the strongest bonds in the silicate structure and can be readily recognized in the infrared spectra of such minerals.

with the thermal product of the clay [23]. The formation of kaolinite during firing above 900°C reduces the amount of free lime and consequently the amount of recarbonated kaolinite. The infrared spectra of the entire fired specimen were compared with the raw samples. The comparisons of the spectra reveal that the bands at 3700-3600 cm^{-1} are clearly modified on firing due to dehydroxylation of kaolinite giving rise to metekaolin as expected. The 1032 cm^{-1} absorption band in the range of asymmetric silicate tetrahedral vibrations are broaden and become shifted to 1092 cm^{-1} due to quartz at above 1100°C further increase of temperature causes increase in the intensity of hematites. It was also found that dehydroxylation of kaolinite between 500°C and 700°C, is accompanied by amorphization since the aluminosilicate sheets are disrupted [20]. The main Si-O stretching band is located at 1032 cm^{-1} in raw materials and increasing firing temperature lead to the slightly displaced toward highest wavenumber (1092 cm^{-1}) reflecting the increasing of Si-O-Si, Al-O-Al bonds along with the formation of new Si-O-Al ones. The characteristics Al-OH liberation mode at 937-912 cm^{-1} lacking. O-H stretching vibrations are highly sensitive to IR radiation. This absorption disappears completlely after heat treatment over 950°C in most of the clay. The lack of OH bands of dehydroxylation occur during calcinations is taken as direct evidence of amorphization

[20]. Si-O vibrations from quartz are more clearly distinguishable as shown in fig, since it remains crystalline in the raw materials. This is observed as the sharp doublet 802-795 cm^{-1}. The broad intense absorption at 1095 cm^{-1}, which due to Si-O stretching in the pure IR spectra is unresolved. These features are also consistent with super position with Si-O vibrations of the SiO_4 condensed tetrahedral of fired clay layer. The overall displacement torward higher energy of the intense band is consistent with reinforcement of the Si-O band of the main phase formed after long firing at1200°C. The enhancement in the broadness of this band at 1088-1099 cm^{-1} torward its lower energy side may be also attributed to anorthite.

Mullite, the Al_2O_3 rich phase of SiO_2-Al_2O_3 system is considered the most important material developed during firing due to the improved thermo-mechanical properties. The sequence of phase transformation departing from amorphous clay aluminosilicate involves formation of Al-Si spinal precursor phase at 1000°C at before crystal mullite. The most easily recognizable of the band at 570 cm^{-1} assignable to Al-O stretching in AlO_6 of the spinal precursor of the mullite phase [21]. Quartz is a residual mineral from the original raw materials, and mullite formed during firing in IFB materials the dehydroxylated kaolin, metakaolin, transforms into a non equilibrium unstable spinal type structure, which converts to mullite above 1000 °C. For the compositions containing by the mixture of kaolin, plastic clay and sawdust materials some modifications in intensity and positions of the bands in the FT-IR spectra indicate the presences of quartz, kaolinite, and hematite. These mineralogical compositions of IFB samples were confirmed with the available literatures.

Conclusion

The FT-IR analysis of insulating fire bricks samples from various compositions of kaolin, plastic clay & sawdust materials indicate the presence of quartz, kaolinite, montmorillonite and hematite. Among the different minerals quartz and kaolinite are present invariably in all samples. Hence, it is considered to be main or major constituents of the samples. FT-IR spectroscopy is one of the major analytical tools. For analysing the both chemical and mineralogical composition of the IFB materials qualitative were performed. Mineralogical study of this IFB samples could be used for various bricks & ceramic industries. The FT-IR analysis results are in good agreement with the ceramic industry standards.

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