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DRIFT- Spectroscopic Study of Modification of Surface Morphology of Perlite During Thermal Activation

KEYWORDS: perlite, DRIFT, thermal activation.

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ABSTRACT *This article presents the use of diffuse reflectance infrared Fourier transform (DRIFT) spectroscopic technique in investigation of the effect of thermal activation on morphology of perlite. Perlite, a naturally occurring waste siliceous material formed by rapid cooling of volcanic eruptions was thermally treated over a range of temperatures. This study focuses on changes in both structure and chemical bonding of perlite due to thermal activation at different temperatures viz., 400, 600, 800, 1000°C for certain time period. The results reveal that on increasing temperature of thermal activation, loss of water occurs which is confirmed by decrease in intensity and broadness of the band, appears between 3600-3300 cm^{-1}, attributing to surface –OH groups. Other bands present in the DRIFT spectra shows the presence of Si-O-Si network and amorphous nature of silica in perlite.*

Introduction

Infrared spectroscopy is a well established technique for the identification of chemical compounds and/or specific functional groups in compounds. An alternative is the use of Fourier Transform Infrared Spectroscopy (FTIR), which is both rapid, non-destructive and requires small, <1 mg, sized samples. Chemical bonds vibrate at a characteristic frequency representative of their structure, bond angle and length. Accordingly, individual molecules have the ability to interact with incident radiation by absorbing the radiation at specific wavelengths. FTIR spectroscopy takes advantage of this by recording the energy absorption of a sample over a range of frequencies. Diffuse reflectance infrared Fourier transform (DRIFT) spectroscopy in conjunction with other analytical techniques has been extensively applied over the years to explore the structure and bonding in amorphous siliceous materials [1]. This technique has proved to be a powerful method to identify the isolated and H-bonded hydroxyl groups on surface of silica [2,3].

R.L. Frost et al. [4] proposed that DRIFT spectroscopy is more applicable than transmission infrared spectroscopy for powdered samples because it provides a rapid technique for analyzing samples without any interference through sample preparation, suitable for the study on the hydroxyl stretching region of silicate minerals. DRIFT has several other advantages including ease of sample preparation, greater number of useful bands and the ability to detect both major and minor components from the same spectra.

Perlite is a hydrated, naturally occurring amorphous volcanic glass formed by cooling of volcanic eruptions, estimating about 700 million tonnes worldwide reserves. Its unique structure consists of numerous concentric layers having SiO_2, Al_2O_3, K_2O and Na_2O as major constituents while TiO_2, CaO, MgO, Fe_2O_3 and hydrated water as well as unburned carbon remain present in varying quantities [5]. On heating the perlite to its softening range, i.e., above 850°C, water molecules vaporize and escape resulting in unusual expansion of perlite up to 7-16 times of its original volume, creating inert, non-toxic, lightweight particles with specific surface area of about 1.22 m^{2}g^{-1} [6], density in the range of 0.6 - 2.30 gml^{-1} [7] and particle size in range of 0.2-4 mm. [8]. As far as applications of perlite are concerned, it is mainly consumed as fillers, filter aids, in producing building construction materials [9,10].

In the current work, perlite was thermally treated at different temperatures viz., 400, 600, 800 and 1000°C for 3 h and then analyzed by DRIFT spectroscopy. The purpose of this investigation is to use DRIFT spectroscopy to characterize the structure and determine the chemical bonding of silica with other species present in the perlite, so that it can be further utilized for various applications in future.

Material

Perlite sample was supplied by Indica Chem. Ind. Pvt. Ltd., India.

Experimental

Perlite sample was thermally treated in a muffle furnace under static conditions over a range of temperatures, 400, 600, 800 and 1000°C for 3h and abbreviated as TAP-400, TAP-600, TAP-800 and TAP-1000 respectively. The DRIFT spectroscopic study of the samples was done by Bruker FT-IR Spectrophotometer (TENSOR 27) in DRS (diffuse reflectance system) mode by homogenizing samples thoroughly with spectroscopic grade KBr in 1:20 weight ratio. The samples were crushed in an agate mortar. The spectra were recorded in the range 550-4000 cm^{-1} with a resolution of 4 cm^{-1}.

Results and discussion

The colour change is seen in perlite from light grey to white-light pink on thermal treatment at higher temperature. The chemical composition of perlite was determined by EDX analysis which is shown in Table 1. Loss on ignition (LOI) was determined by heating a certain weighed quantity of perlite in muffle furnace at 1000˚C for 3 h. The LOI amount was 4.4 wt % which corresponds to the removal of moisture and coexisting unburned carbon from sample [11].

Table 1. EDX analysis of perlite.

Samples	O(wt%)	Si(wt%)	Al(wt%)	K(wt%)	Na(wt%)	Zn(wt%)	Fe(wt%)	Ti(wt%)	S(wt%)	LOI
Perlite	73.70	18.83	3.72	1.44	1.91	0.22	0.10	0.07	-	4.4

LOI- Loss on ignition

The FT-IR spectra of perlite and thermally activated perlite at different temperatures confirmed that the calcination at any conditions in this experiment produced dehydroxylation in the perlite samples i.e. elimination of the -OH stretching from Si-OH [12,13]. In this fig., a broad band between 3600-3300 cm^{-1} is shown, which is attributed to surface –OH groups of –Si-OH and water molecules adsorbed on the surface. The broadness of band indicates the existence of hydroxyl groups in higher degree of association with each other which results in extensive hydrogen bonding [14], while in FT-IR spectra of thermally activated perlite samples, the intensity and broadness of band is decreased, confirming the loss of water, which is highest in case of TAP-1000 (Fig. 1e). The strong band at 1030 cm^{-1} is due to the structural siloxane framework, which is the vibrational frequency of the Si-O-Si bond. The peak gets shifted to higher wave number, i.e., 1227 cm^{-1} after thermal treatment in TAP-1000 (fig. 1e), normally observed in amorphous silica samples [15]. An intense peak at 1632 cm^{-1} in the spectrum of perlite is attributed to bending mode ($\delta_{O\text{-}H}$) of water molecule, which is again highly decreased in case of TAP-1000. The shoulder at about 3200 cm^{-1} (fig. 1a) could be assigned to the stretching vibrations of Si-OH groups in the structure of amorphous SiO_2 [16]. An intense band in the range of 1300-1100 cm^{-1}, corresponding to valence vibrations of the silicate oxygen skeleton is usually assigned to the amorphous silica content. The region around 805 cm^{-1} is characteristic of Si-O-Si symmetric stretching modes [17,18,19]. In the Si–O stretching vibration region (800–1195 cm^{-1}), the bands at 802, 808, 812, 942, 1050 cm^{-1} are identical to the bands at 800, 958, 1088 cm–1 due to amorphous silica [20]. Amorphous silica exhibited a relatively strong peak at about 800 cm^{-1} and it can be distinguished from the band of crystalline silicate [20]. The structure of most SiO_2 is polymorphous, both crystalline and amorphous, based on tetrahedral unit of silicon coordinated to four oxygen atoms. In the Si–O–Si bending vibration region (400–700 cm^{-1}) of quartz, the band at 695 cm^{-1} is determinative whether it is crystalline or amorphous [21]. The band at 695 cm^{-1} appears due to the vibrations in octahedral site symmetry [22]. In the amorphous state this band will be missing. In the perlite samples, we did not get this band which indicates that the silica mineral in this sample is in amorphous form. Another evidence of the presence of amorphous silica in perlite sample is the appearance of a peak at about 1100 cm^{-1}, which is normally assumed to be formed by continuous network of Q^4 species, characteristic in case of amorphous silica [23].

The major component of perlite is silica and untreated silica is totally hydroxylated and the hydroxyl layer is covered with physically adsorbed water. Thermal treatment of the support leads first to removal of water (dehydration) and then to combination of adjacent hydroxyl groups to form water (dehydroxylation) [24].

Fig. 2 shows the magnified FT-IR spectra of all studied samples in the range between 550-1750 cm^{-1}.

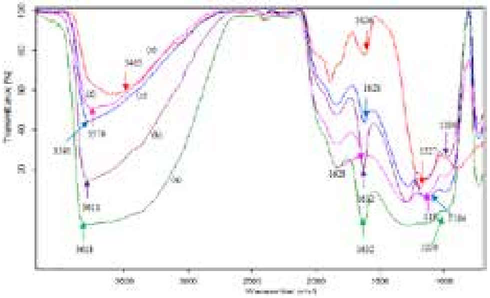

Fig 1. FT-IR spectrum of (a) perlite, (b) TAP-400, (c) TAP-600, (d) TAP-800 and (e) TAP-1000.

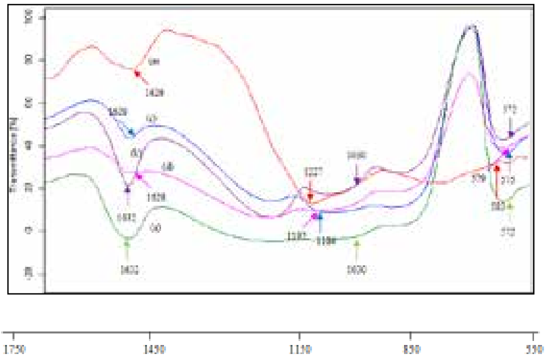

Fig 2. Magnified FT-IR spectrum of (a) perlite (b) TAP-400 (c) TAP-600 (d) TAP-800 and (e) TAP-1000

The observed frequencies of IR bands of all samples and their possible assignments are summarized in Table 2.

Table 2. The observed frequencies of IR Bands of all samples and their possible assignments

Assignments		Wave number (cm^{-1})			
	Perlite	TAP-400	TAP-600	TAP-800	TAP-1000
Si-O-Si symm. stretching vib.	802	802	808	812	942
Si-O-Si asymm. stretching vib.	1030	1030	1184	1192 1192	1227
Si-O-Si bending vib.	572	572	575	579	583
-O-H bending vib.	1632	1632	1628	1628	1626 1626
-O-H stretching vib.	3618	3611	3591	3579	3465

Conclusions

Results found in this paper showed that DRIFT can be successfully employed in investigation of the effect of thermal treatment on perlite morphology. After calcination, physically adsorbed water gets driven away from the surface, so intensity of –OH stretching and bending bands decreases. The present spectroscopic study could be useful in understanding characteristic features of perlite, changes occurred in its structure and chemical bonding because of thermal treatment. This knowledge may be employed in further utilization of perlite for several applications.

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