

Pyrophyllite as raw material for ceramic applications in the perspective of its pyro-chemical properties

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Abstract

General pyro-chemical properties of pyrophyllite were studied using one Indian variety as sample. In addition to normal routine analysis, thermal expansion, infrared spectroscopy, DTA, XRD and SEM studies were also employed to understand the pyro-chemical properties of the specimen at different temperatures. The results indicate that the specimen contains pyrophyllite as major phase with sericite, quartz and diaspore as minor phases. Unlike kaolinite, pyrophyllite contains low alumina and high silica which on heating mainly produces mullite and amorphous silica. Mullite crystallization from pyrophyllite is rather easy than that from kaolinite. The silica in turn yields large amount of viscous liquid at high temperature. It is suggested that pyrophyllite may be utilized in such compositions favourably where mullite is a desirable phase by partial replacement of china clay which is a viable alternative particularly in the background of depleting reserves of kaolinite and its continuous cost escalation. Additionally amorphous silica produced in the reaction system may acts as an in situ produced filler material that reduces the use of quartz in such system.

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1. Introduction

Pyrophyllite is a chemically inert hydrous aluminosilicate $\text{Al}_2\text{O}_3 \cdot 4\text{SiO}_2 \cdot \text{H}_2\text{O}$ [$\text{Al}_2\text{Si}_4\text{O}_{10}(\text{OH})_2$], composed of 67% SiO_2 , 28% Al_2O_3 and 5% H_2O . It is an early stage metamorphic mineral and is actually quite common. Pyrophyllite is largely an overlooked raw material available in huge quantities in nature. This is mainly due to the fact that other comparable aluminosilicate raw materials like china clay are available in much purer form.

Huge reserves of pyrophyllite is found in various parts of the globe among which the most important locations are USA, New Zealand, China, South Africa, Australia, Brazil, etc. In India, pyrophyllite mineral is found mainly in the states of Uttar Pradesh, Madhya Pradesh and Maharashtra [1,2]. An estimated reserve of 1.0 million tons of pyrophyllite is available only in Maharashtra. The mineral is extensively used as filler in rubber, paper, soap, cosmetics, asbestos products and insecticides;

however, use of pyrophyllite in ceramic industries is very much restricted.

Studies on pyrophyllite have been carried out by several investigators throughout the world, although its use as a ceramic raw material is not yet widely accepted. This is probably due to the fact that the mineral seldom occurs in pure state like kaolinite and the characteristics of associated minerals (quartz, kyanite, sericite, diaspore) can influence its properties and behaviour. Literature indicates that most investigators studied the properties of pyrophyllite and its utilization based on well-known sources, i.e. pyrophyllite from Hillsboro, NC, USA [3], Coromandel Peninsula, New Zealand [4], and from coastal region of Southeast China [5].

Most of the investigations on pyrophyllite were confined to its utilization in whiteware body as a replacement of quartz, clay or feldspar [6–15]. However, there is little information regarding thermal transformation of pyrophyllite mineral. Bradley and Grim [16] and later Heller [17] studied the thermal transformation of pyrophyllite to mullite. Nakahira and Kato [18] also studied the thermal reactions of pyrophyllite by transmission electron microscopy (TEM). The investigations were carried out to evaluate the crystallographic structure of

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pyrophyllite and its dehydroxylate. Kersey and Jerner [19] studied the mechanical properties of pyrophyllite as a function of firing temperature up to 1050 °C in air as well as in hydrogen atmosphere and tried to correlate the properties with microstructure. Sanchez-Soto et al. [20] studied the effect of mechanical treatment on pyrophyllite followed by thermal treatment. They concluded that there was possible segregation of amorphous silica and that mullite formation was rapid at lower temperature. Scanning electron microscope (SEM) studies were used by the same researchers [21] to understand different morphologies developed when pyrophyllite was progressively transformed by heating. However, very little information about the thermal transformations of pyrophyllite over a range of temperatures has been published with particular emphasis on thermo-mechanical properties as well as the content and crystallinity of mullite.

In our earlier communications one particular variety of pyrophyllite from Maharashtra was incorporated in a triaxial porcelain composition as a replacement of quartz and china clay [22–24] and the effect on the vitrification, mechanical properties and microstructure was studied. It was observed that pyrophyllite could be used in the tune of 15–22% as a replacement of quartz and china clay. In continuation to the same study, pyro-chemical properties of pyrophyllite mineral was evaluated and analyzed to understand the compatibility of the mineral, in general, during use in combination with other constituents in the triaxial porcelain system. The investigation will open up avenues to understand the mineral and its subsequent utilization due to depleting resources of standard alumino-silicate minerals.

2. Experimental

The sample of pyrophyllite was obtained from Maharashtra, India. Standard methods of coning and quartering were adopted for obtaining representative samples from the bulk. Specific gravity of the sample was determined with (–)200 mesh powder following usual procedure [25] using specific gravity bottle.

The pyrophyllite sample was ground to 300 mesh and then mixed with 1% poly vinyl alcohol (PVA) solution to obtain a mix with moisture content in the range of 6–8%. Discs of about 50 mm diameter and 10 mm thickness were pressed at 250 kg cm⁻² in a semi-automatic hydraulic press. The discs were initially dried at room temperature to avoid cracking. The air dried discs were then finally dried in an electric oven by gradual rise of temperature up to 110 °C in 6 h and left for overnight. The samples were then cooled in the desiccators and their diameter measured to determine the dry linear shrinkage. Average of five samples was reported as the measured value. Dry test specimens were fired in an electrically heated muffle furnace at six different temperatures between 1100 °C and 1350 °C with 50 °C interval and 2 h soaking time at respective peak temperatures. Fired linear shrinkages, water absorption and bulk density of the fired specimens were determined following the standard procedure [26].

Thermal expansion of green as well as fired (1200 °C) pyrophyllite samples was investigated by an Orton Automatic Dilatometer, Bosch & Lamb, UK make, and at a heating rate of 2 K/min. Green samples were prepared by cutting raw pyrophyllite with diamond wheel followed by grinding and polishing. DTA, X-ray diffraction and chemical analyses of the samples were carried out for identification of possible mineral phases present. Chemical analysis was carried out following wet chemical as well as instrumental analysis. SiO₂ was estimated gravimetrically [27], Na₂O and K₂O flame photometrically [28] and other oxide constituents were estimated [29] using Inductively coupled plasma atomic emission spectrometer, Spectro Analytical Instrument, Spectro Ciros Vision model, Germany. DTA was performed using Netzsch Thermal Analyzer, model STA 409 with DTG mode determining DTA and TG simultaneously. Both DTA and TG of the samples were studied under identical conditions up to 1200 °C at a heating rate of 10 °C/min in alumina crucible and using α -alumina as the reference. The X-ray diffraction studies were carried out with powder (–200 mesh BS sieve) of each of the samples. ‘X’Pert Pro’ MPD diffractometer (PANalytical) attached with secondary monochromator, automatic divergence slit and nickel filter was used and operating at 45 kV and 40 mA to get monochromatic Cu-K α radiation. The instrument was run at step scan mode with step size (0.02) and 8 s time per step, with 2θ angle 10–60°. The collected data was refined using Profit software. X-Pert plus and Quasor software based on Rietveld were used to calculate the percentage of mullite and quartz, where standard quartz and mullite were used as reference materials. The weight percentage of glassy phase was estimated by subtracting sum of the wt% of quartz and mullite from 100. For reliability of data, each sample was scanned several times. The fitted curve matched well with the raw data and ‘goodness of fit’ varied from 2.5 to 4.0 among different samples. Microstructure was studied by SEM analysis on powdered raw pyrophyllite as well as on some selected sintered samples using a LEO S-430i apparatus. For SEM studies on fired specimens, the specimens were polished to 1 μ m finish with diamond paste after initial grinding with Silicon carbide powder/Cerium oxide powder and water. The polished surfaces of such samples were etched with 5% HF solution for 60 s duration at room temperature (35 °C), washed with distilled water and acetone followed by gold sputter coating (Edwards, Scancoat).

3. Results and discussion

The sample of pyrophyllite was obtained as grayish white hard massive lumps. There were occasional green and purple patches in the as received lumps. The specific gravity of the as received sample was 2.743. Dry linear shrinkage was 2.33% (± 0.12).

The chemical analysis of the pyrophyllite sample has been presented in Table 1. The results indicate that the sample was rather low in SiO₂ (59.51%) and rich in Al₂O₃ (30.43%) in comparison to those of theoretical values (SiO₂ 67% and Al₂O₃ 28%). The presence of diasporite as indicated by other studies

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