

STUDIES ON THERMAL OXIDATION OF CHALCOPYRITE FROM CHITRADURGA, KARNATAKA STATE, INDIA

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When chalcopyrite is heated in air, up to 350^o there is no marked change. Between 350 and 440^o, surface material is oxidised to iron sulphate, CuSO₄ and Fe₂O₃, while in regions not accessible to oxygen the formation of Cu₅FeS₄, FeS and S takes place. From 440 to 500^o oxidation and sulphation phenomena occur. Stable compounds between 500 and 650^o are iron sulphate, CuSO₄ and Fe₂O₃, with a minor amount of 6CuO.Cu₂O indicated at 650^o. After the decomposition of iron sulphate, CuSO₄ decomposes, first to CuO.CuSO₄ and then to CuO. By 750^o the sulphur has been totally lost from all compounds, while the oxides of copper and iron partly react to form CuFe₂O₄. Final products of oxidation between 800 and 850^o are CuO, CuFe₂O₄ and Fe₃O₄.

The mechanism of thermal oxidation of chalcopyrite (CuFeS₂) in air has been examined in detail by many workers [1]. An attempt has been made in this paper to identify the products and to study the process of thermal oxidation of chalcopyrite in air, so as to establish a sequential relationship for the compounds formed when the mineral is progressively heated up to 850^o. Techniques employed in this study are DTA, TG, X-ray diffractometry and colorimetry.

Experimental

Material

The chalcopyrite concentrates from Chitradurga, Karnataka State in South India selected for the present work contain 25% Cu, 27.90% Fe, 26.92% S, 0.75% Pb and 0.15% Zn. Non-sulphide impurities in minor amounts are CaO, MgO, Al₂O₃ and SiO₂.

Thermal analysis

DTA: Leeds and Northrup Unit provided with Pt-Pt/10% Rh thermocouples and a Robert Grim-Shaw type ceramic sample holder was used, with calcined alumina as the thermally inert reference material. The rate of heating was 2 deg per minute.

TG: The sample was heated in a platinum bucket hooked to the pan of a Mettler balance and suspended in a vertical tubular furnace of length 25 cm and diameter 2 cm. The thermocouple (Pt-Pt/13%Rh) for recording the temperature of the sample was

kept very close to the bucket. The rate of heating was 2 deg per minute. Weights and temperatures of the sample were recorded simultaneously at intervals of 5 minutes.

XRD

X-ray diffractometer traces were taken on a Philips PW 1010 diffractometer with a Nonius-Guinier camera 11.46 cm in diameter, using Cu K alpha radiation.

Sample preparation for XRD studies

(a) A vertical tubular furnace 25 cm long and 5 cm in diameter, plugged at both ends with ceramic blocks, was used for roasting the samples. A 3 mm hole was provided in the plugs at both ends for inlet of the thermocouple and a natural draught of air. A 3 g sample (average particle size 44 μm), with ceramic beads distributed uniformly in the mass of the sample for easy and uniform accessibility of air, was taken in a Gooch crucible (3.5 cm diameter and 4.5 cm height). The crucible, placed over a ceramic tripod, was kept on the ceramic plug at the bottom of the furnace. The furnace temperature was maintained at the desired value for 3 hours to ensure the formation of maximum amounts of the compounds expected at each temperature. (b) To obtain a clear XRD pattern of water-insoluble compounds formed during roasting, the masking effect of water-soluble salts was removed as follows: One g of the sample prepared as in (a) above was boiled in 20 ml distilled water for 15 minutes, after which the mixture was centrifuged and decanted. This process was repeated 5 times, after which the solids were dried, and the washings were stored for chemical analysis.

Samples thus prepared were stored in a desiccator and later subjected to X-ray analysis.

Chemical analyses

Water-soluble copper and iron in the washings obtained in (b) above were determined quantitatively by colorimetry.

Results

DTA: The DTA curve of the natural sample (Fig. 1a) shows an exothermic drift up to about 330^o after which there is a series of 4 peaks on the exothermic side of the baseline till 545^o. The baseline of the curve between 545 and 675^o is straight, indicating this to be a region of equilibrium. Finally, in the temperature interval 675 to 780^o there is a well marked double endothermic peak.

TG: The TG curve (Fig. 1b) initially shows a gradual loss in weight, this reaching a maximum of about 7% at 350^o. This is followed by a continuous gain in weight with a maximum of about 20% at 540^o. Between 540 and 620^o there is a slight fluctuation in weight, after which there is a rapid loss in weight, which reaches a maximum of about 25% at 800^o. Between 800 and 900^o the changes in weight are not appreciable.