PROCEEDINGS: PROF–97
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Effect of acid leaching on recovery of graphite from calcareous deposits

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ABSTRACT

In this paper an attempt has been made for optimisation of acid leaching of calcite from graphite ore of Tamilnadu. The ore containing 39% acid soluble and 61% insolubles. The best leaching parameters obtained at particle size below 150 microns are reaction time 20 minutes, temperature 40°C, solid liquid ratio 1:4 and stoichiometric ratio of acid 0.69. Flotation test results on partial leached and on unleached sample indicate that the graphite grade and recoveries are improved from a calcite leached sample. The flotation product obtained from a leached sample contains 73% fixed carbon with 83% recovery.

INTRODUCTION

Graphite deposits occur all over the country mostly either as flaky or amorphous. Association of graphite with siliceous deposits are quiet common than calcareous deposits. In India there are two calcareous graphite deposits found at (a) Rajasthan and (b) Tamilnadu[1]. These deposits are being exploited for industries after beneficiation. Since the flotation products contain high amount of calcium, these products are not widely marketable. Literature indicate that graphite is floated after pretreatments such as calcination or leaching[2-5]. However, there is no systematic approach on recovery of graphite from calcareous deposits. Hence in this paper an attempt has been made for optimisation of acid leaching and few flotation tests on calcareous graphite ore of Tamilnadu.

MATERIALS AND METHODS

Graphite sample was procured from M/s. Saak Trading Company, Madurai, Tamilnadu, containing 11.0% fixed carbon. Representative sample of –100 micron size fraction was prepared by stage crushing, grinding and screening. Mineralogical identification was carried out by using Philips X-ray diffractometer. Leaching

of calcium carbonate was carried out by using analar grade hydrochloric acid in a glass reactor. The variables maintained were acid strength, temperature, solid liquid ratio and reaction time. Leach liquors of these samples were filtered and analysed for calcium by wet chemical analysis. Flotation studies were carried out on partial leached sample and also on original sample. For flotation tests, sodium silicate as depressant, 0.1 kg/t, pine oil as forther, 1.5 kg/t, were used. The conditioning was done at 40% solids concentration and floated the graphite at 10% solids concentration. The concentrate and tailing of each flotation test was analysed for fixed carbon (FC).

RESULTS AND DISCUSSION

The size, fixed carbon and distribution of calcium oxide in the crushed ore sample are shown in Fig. 1. The data indicate that the graphite is equally distributed at all size fractions where as the calcite is more concentrated at above 0.50 mm and at below 0.15 mm size. Typical X-ray diffraction data shown in Fig. 2, confirm the presence of graphite, calcite and quartz minerals in the ore.

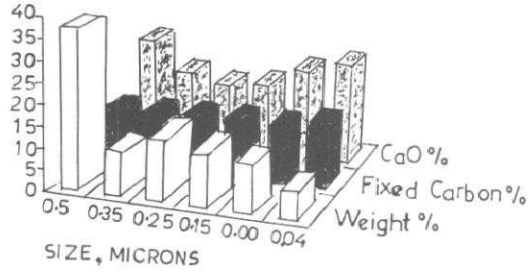

Fig 1 : The distribution of size, FC and CaO in natural graphite sample.

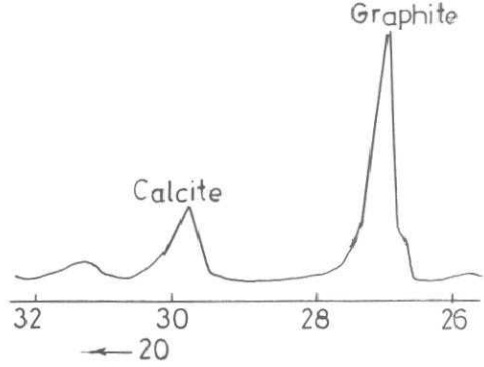

Fig. 2 : X-ray diffraction pattern of graphite samples.

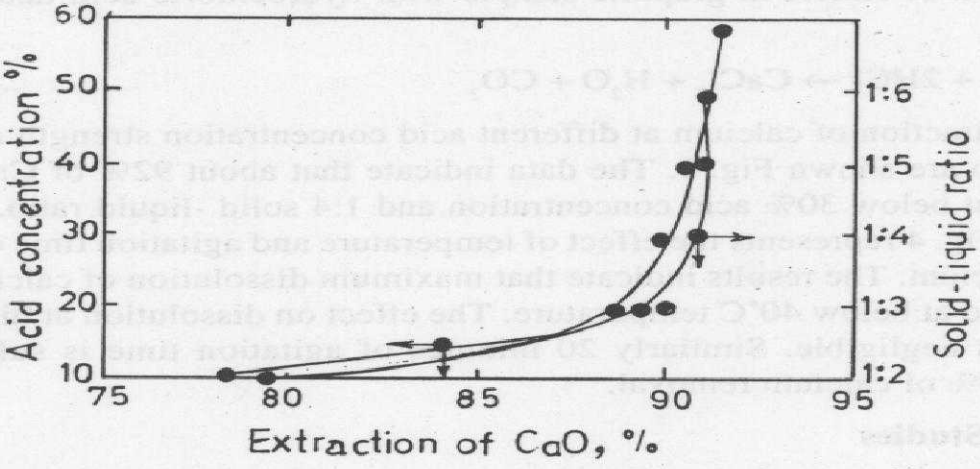

Fig 3 : Effect of acid concentration and solid-liquid ratio on extraction of CaO.

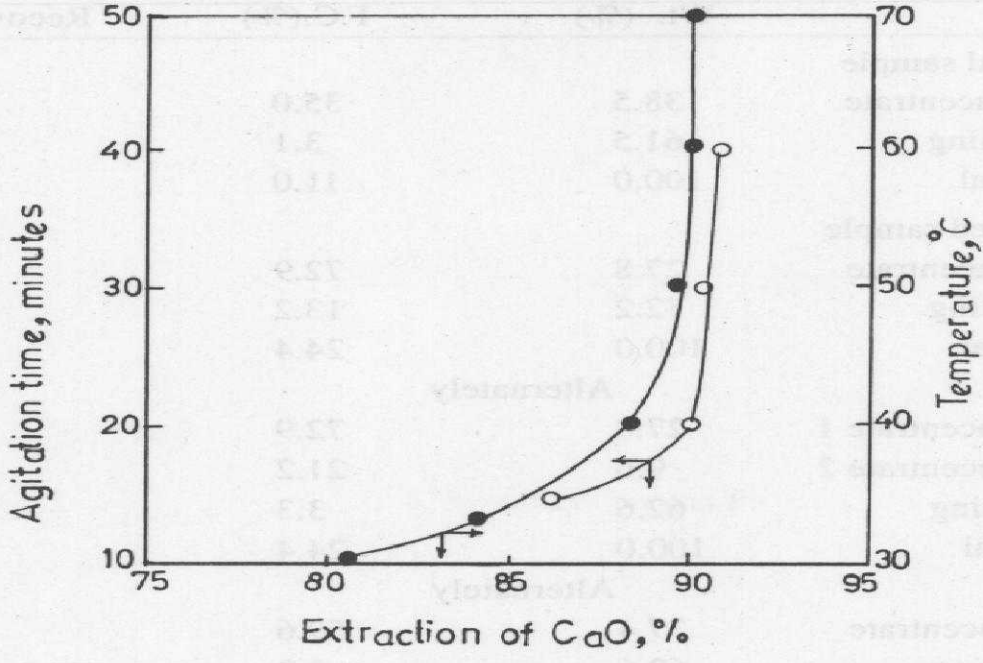

Fig 4 : Effect of temperature and agitation time on extraction of CaO.

Dissolution studies

Reaction of calcite in graphite sample with hydrochloric acid take place as follows :

$$CaCO_3 + 2HCl \rightarrow CaCl_2 + H_2O + CO_2 \quad \ldots (1)$$

The extraction of calcium at different acid concentration strength and solid-liquid ratio are shown Fig. 3. The data indicate that about 92% of CaO can be extracted at below 30% acid concentration and 1:4 solid -liquid ratio. The data shown in Fig. 4 represents the effect of temperature and agitation time on extraction of calcium. The results indicate that maximum dissolution of calcium could be achieved at below 40°C temperature. The effect on dissolution at higher temperature is negligible. Similarly 20 minutes of agitation time is sufficient to achieve 90% of calcium removal.

Flotation Studies

The data shown in Table 1, represents the flotation results on leached and natural samples. From this data it is seen that a rougher concentrate containing

Table 1 : Comparison of flotation test results on natural and acid leached samples

Details	Wt. (%)	F.C.(%)	Recovery (%)
a) Natural sample			
Concentrate	38.5	35.0	88
Tailing	61.5	3.1	12
Total	100.0	11.0	100
b) Leached sample			
Concentrate	27.8	72.9	83
Tailing	72.2	13.2	17
Total	100.0	24.4	100
	Alternately		
Concentrate 1	27.8	72.9	83
Concentrate 2	9.6	21.2	8
Tailing	62.6	3.3	9
Total	100.0	24.4	100
	Alternately		
Concentrate	37.4	59.6	91
Tailing	62.6	3.3	9
Total	100.0	24.4	100

35% F.C. with 88% recovery could be achieved by flotation of natural sample. But in case of leached sample, it is 59.6 % FC with 91 % recovery. However, the tailings of both the leached and natural samples contain 3% fixed carbon only. It is also possible to obtain 73% FC from the same flotation operation test on leached sample. But the recovery is 83%. The middling contain 21% FC has to be recirculated and rejection of the tailings with 3% FC.

CONCLUSIONS

The graphite ore sample contained 39% acid soluble (major calcite mineral), and 61% acid insolubles (quartz, graphite etc.). The best leaching parameters obtained at particle size below 150 microns are: temperature-40°C, reaction time-20 minutes, solid-liquid ratio : 1:4 and stoichiometric ratio of acid 0.69. Flotation results of natural and partial by leached graphite samples resulted in concentrates assaying 35% F.C., with 88% recovery and 59.6% F.C., with 91% recovery respectively. Alternately at 83% recovery a product containing 73% FC could be achieved from a leached sample. However, it needs further studies on beneficiation.

ACKNOWLEDGEMENTS

The authors are thankful to Prof. H.S. Ray, Director, Regional Research Laboratory for permitting to publish this paper. One of the authors Ms. Nivedita is thankful to Mr. B. C. Mohanty, Regional Research Laboratory and Mr. Patil is thankful to Prof. K. S. Sivakumar, Gulbarga University for the encouragement to carry out the work.

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