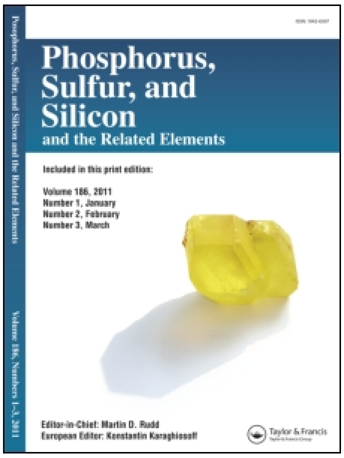

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Recovery of Phosphate from Steel Manufacture Slag by Sulfuric Acid Treatment

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RECOVERY OF PHOSPHATE FROM STEEL MANUFACTURE SLAG BY SULFURIC ACID TREATMENT

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The sulfuric acid treatment on steel manufacture slag (SMS) was performed to recover P resource. P element in SMS was successfully removed. Final products were a solid of $CaSO_4/SiO_2$ and a solution containing PO_4^{3-}, Fe, Al, Mg, and Mn.

Keywords: $CaSO_4$; P; recovering; slag; sulfuric acid; steel manufacture

INTRODUCTION

All of P resource has been imported from other countries to Japan;[1] however, there are no recovering processes in industries. On the other hand, free lime containing a lot of P resource produced from steal manufacture processes has not been utilized as cement raw materials or road board materials because of high content of P in free lime.[1] Most of such free lime has been accumulated in industrial farm. Therefore, we have to utilize such a free lime in industry and recover P resource in free lime as recycled P resource. For this purpose, P element should be removed from free lime produced in steel manufacture industry. In some patents, a few noble processes have been proposed.[2] In this study, a sulfuric acid leaching process was investigated in order to eliminate P from steel manufacture slag (SMS) produced in a steel industry process.

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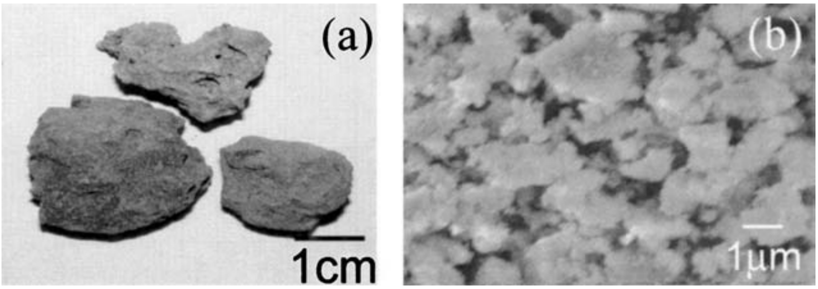

FIGURE 1 Photograph (a) and scanning electron micrograph (b) of SMS.

EXPERIMENTAL

SMS was obtained from a steel company. This is a block in which size is a few centimeters. This block consists of particles with 1 μm diameter. Photograph and electron micrograph are shown in Figure 1. Chemical composition of SMS was determined by Energy Dispersible X-ray (EDX) analysis. The obtained chemical compositions were summarized in Table I. A content of P was estimated to be 6.1 weight %. This P provides a big problem when using SMS as cement raw material and road board material.

Figure 2 shows a scheme of a sulfuric acid treatment on SMS used in this study. Various H_2SO_4 concentrations were used, and reactions took place at 20°C. After the treatment, a solution was analyzed by Inductively Coupled Plasma (ICP) absorption spectroscopy, and a solid was analyzed with an x-ray diffraction method. Moreover, a surface area of solid was also determined with BET.

RESULTS AND DISCUSSION

Figure 3 shows the chemical composition changes of solid after the sulfuric acid treatment (1.0 mol dm^{-3}) at 20°C. These chemical compositions were obtained with EDX analysis. The Ca and Si contents did not significantly change during the sulfuric acid treatment, but the S content was increased rapidly during the initial 1 h. Other elements were sharply decreased. Only iron ions were slowly decreased. These results

TABLE I Chemical Composition of SMS used in this Study

Element	Mg	Al	Si	P	S	Ca	Mn	Fe	Others
wt%	5.1	7.6	10.8	6.1	0	51.2	3.4	15.2	0.6

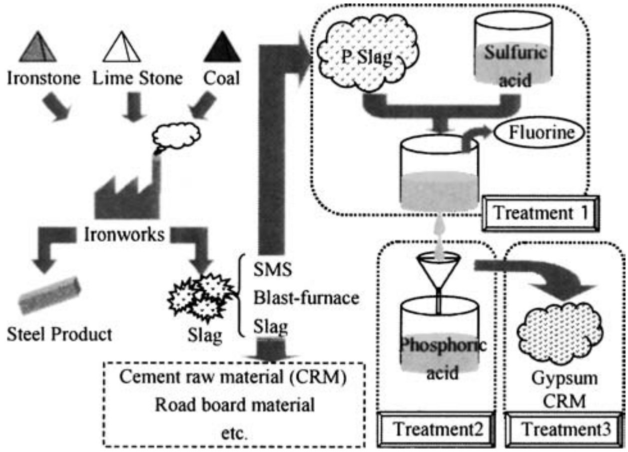

FIGURE 2 Scheme of the sulfuric acid treatment on SMS proposed in this study.

indicate that P element in SMS can be removed by the sulfuric acid treatment. Simultaneously, sulfate ion enters into the solid phase. In other words, an ion exchange between PO_4^{3-} ions and SO_4^{2-} ion occurs during the immersion of SMS in sulfuric acid solution.

Figure 4 shows the x-ray diffraction patterns of SMS after the sulfuric acid treatment (1.0 mol dm^{-3}). SMS before the treatment was

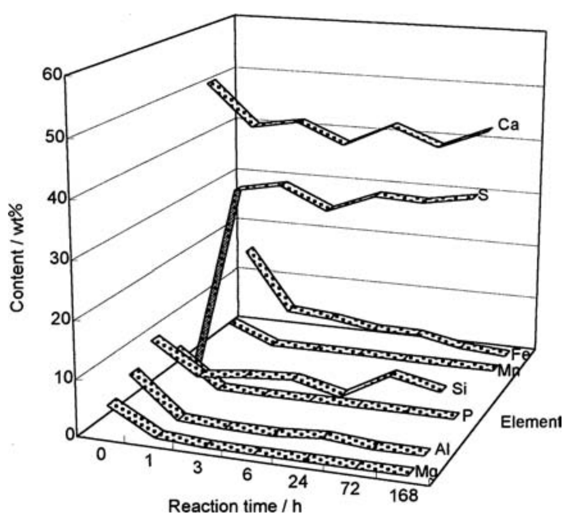

FIGURE 3 Chemical composition changes for each element during the sulfuric acid treatment (1.0 mol dm^{-3}).

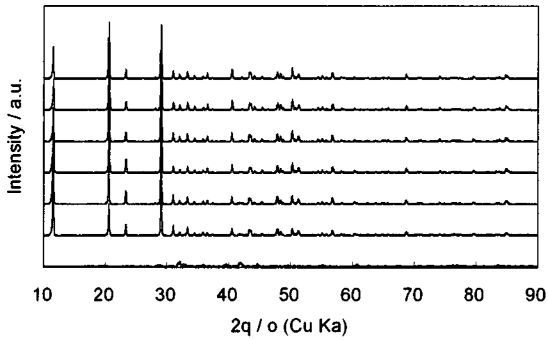

FIGURE 4 X-ray diffraction patterns of SMS after the sulfuric acid treatment at various reaction durations.

almost amorphous. After 1 h treatment, sharp peaks were observed and were assigned to $CaSO_4{\cdot}2H_2O$. During the treatment for 168 h, no changes were observed in these x-ray diffraction patterns, indicating that the ion exchange reaction was almost terminated within 1 h. As a result of the ion exchange, amorphous Ca-Si-P was converted to $CaSO_4{\cdot}2H_2O$. When using 5.0 mol dm^{-3} H_2SO_4, a similar result was obtained. The surface area of $CaSO_4{\cdot}2H_2O$ is generally important for hydration of $CaSO_4{\cdot}2H_2O$ particles. The surface area of $CaSO_4{\cdot}2H_2O$ obtained using two different H_2SO_4 concentrations was measured with BET. $CaSO_4{\cdot}2H_2O$ produced in 1.0 mol dm^{-3} H_2SO_4 was 26 $m^2\,g^{-1}$, and that produced in 5.0 mol dm^{-3} H_2SO_4 was 60 $m^2\,g^{-1}$. $CaSO_4{\cdot}2H_2O$ was heated at 140°C for 3 h to eliminate H_2O and to form $CaSO_4{\cdot}1/2H_2O$. A hydration of $CaSO_4{\cdot}1/2H_2O$ was conducted at water/solid ratio = 1:1 for 24 h. The hydration process of the prepared $CaSO_4{\cdot}1/2H_2O$ was similar to that of reagent; however, a small amount of iron ions remained in the prepared sample so that a color of hydrate was not completely white.

CONCLUSION

The sulfuric acid treatment was investigated in order to remove P on SMS. During the immersion of SMS in sulfuric acid solution, the following reactions occur:

$$\begin{aligned} \text{SMS} + H_2SO_4 &\rightarrow CaSO_4{\cdot}2H_2O - SiO_2 \text{ (solid phase)} \\ &\quad + H_3PO_4 + HF + \text{ other elements (solution phase).} \end{aligned}$$

H_3PO_4 ion, HF, Al, Mg, Mn, and Fe are involved in the solution phase, and Ca, Si, SO_4^{2-}, and a small amount of Fe are involved in the solid phase. Thus, the simple sulfuric acid treatment is effective to recover P element from SMS.

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