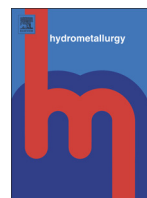




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Control of silica polymerisation during ferromanganese slag sulphuric acid digestion and water leaching

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ABSTRACT

A major obstacle to the hydrometallurgical treatment of ferromanganese slags is the way in which silica polymerisation is controlled during sulphuric acid digestion and water leaching. In an acidic medium, silica enters in solution and forms silicic acid, which polymerises into silica gel and makes solid-liquid separation difficult.

This article will show that the quick leach model, also called the water-starved system, limits silica solubilisation and its subsequent polymerisation during manganese extraction from ferromanganese slags by rejecting most of the ferromanganese slag silica content in the leach residue. It will further illustrate that the silica content in the residue is dependent on the acid concentration used during acid digestion. It will also show that dissolved silica in the pregnant leach solution is high when an unrestricted quantity of water is used and low when the water quantity is restricted during water leaching. Furthermore, a method for silica gel recovery from ferromanganese slag will be presented, as well as the silica gel analysis, in order to confirm the theory presented in this article.

Manganese extraction of up to 90% is obtained and the leach residue presents good latent hydraulic properties, which can be used as an addition to Portland cement or a gypsum replacement. More than 95% of the initial silica content of the slag is rejected in the leach residue, and a residue silica content of more than 27% is obtained. Thus, silica solubilisation and polymerisation are controlled and solid-liquid separation is accelerated.

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1. Introduction

Ferromanganese slags contain silicate phases. The covalent bond between silicon and oxygen in silicates is a strong bond that cannot be easily broken by aqueous solutions (Habashi, 1993). The destruction of silicate structures by acid occurs as a result of the dissolution of metals present in the structure. This dissolution results in the collapse of the three-dimensional silicate structure to form silicic acid (Parris, 2009).

Silicate minerals attacked with acid either gelatinise or separate into insoluble silica. Those which gelatinise are (Murata, 1943):

- Those containing silicate radicals of small molecular weight, and possibly silicates containing ring structures of three silicon atoms (such as orthosilicates, pyrosilicates);
- Those containing large continuous silicon-oxygen networks (disilicates containing appreciable ferric iron in the silicon-oxygen sheets, and minerals of the silica type with three-dimensional networks that contain aluminium in a ratio of at least two aluminium atoms to three silicon atoms); and

- Those which separate into insoluble silica are characterised by silicon-oxygen structures of large dimensions that do not disintegrate into small units under acid attack, such as SiO_8 chains, SiO_{11} chains, and Si_2O_5 sheets that do not contain large amounts of ferric iron that replace silicon, and three-dimensional networks that have an aluminium content lower than the ratio of two aluminium atoms to three silicon (Murata, 1943).

In order to solubilise valuable silicates for metal recovery, a thermal treatment is usually required before leaching (Habashi, 1993).

Silica polymerisation is a major problem during ferromanganese slag acid leaching as it makes filtration difficult afterward. This article presents techniques on how to deal with silica polymerisation, and uses the quick leach or water-starved model described by Dufresne (1976). This model is used to minimise silica polymerisation, and concentrated sulphuric acid is used because of its ability to destroy the structure of the silicate phase by dissolving metals in the structure.

In the available literature, little is reported in terms of work focused on the leaching of ferromanganese slags in sulphuric acid in order to recover manganese values and to limit silica polymerisation. Past investigations are described elsewhere (Kazadi et al., 2013). Of great interest to this study is the work of Das et al. (1978). Das et al. did some leaching

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work on ferromanganese slags using hydrochloric acid and sulphuric acid at different temperatures, ranging from room temperature to 99 °C. Silica gel polymerisation was observed as being a major problem during filtration. The effects of acid quantity, temperature, leaching duration, solid-liquid ratio and particle size, were studied. An extraction of 90% manganese was achieved, but silica polymerisation is not addressed.

2. Silica polymerisation

Silica has different degrees of crystallisation, exhibiting different solubilities in aqueous solutions. The highest degree of solubility is exhibited by amorphous silica (Queneau and Berthold, 1986) – the form of silica which is precipitated from aqueous solution and which is also present in the leach residue of ferromanganese slags (Kazadi et al., 2013).

Thermodynamically, in acidic solution, soluble silica is present as the monomer silicic acid $\text{Si}(\text{OH})_4$, as seen in the Eh–pH diagram of silica in water (Fig. 1). Fig. 2 provides the solubility of amorphous silica in aqueous solution as a function of pH. In practice, 100 to 200 mg/l as SiO_2 is assumed to be the solubility range of amorphous silica in acidic medium (depending on particle size, state of internal hydration, and the presence of impurities absorbed in the silica or on its surface) (Iler, 1955). However, in acidic solution, silica concentrations in excess of the equilibrium solubility of monomeric silica are encountered, which is also known as supersaturated solution. The ageing of these solutions results in the formation of amorphous silica, either in the form of a colloid or a gel, called silica gel (Queneau and Berthold, 1986). It must also be noted that a supersaturated silica solution can remain crystal clear for a long period of time due to the silica gel's optical properties in solution (Parris, 2009).

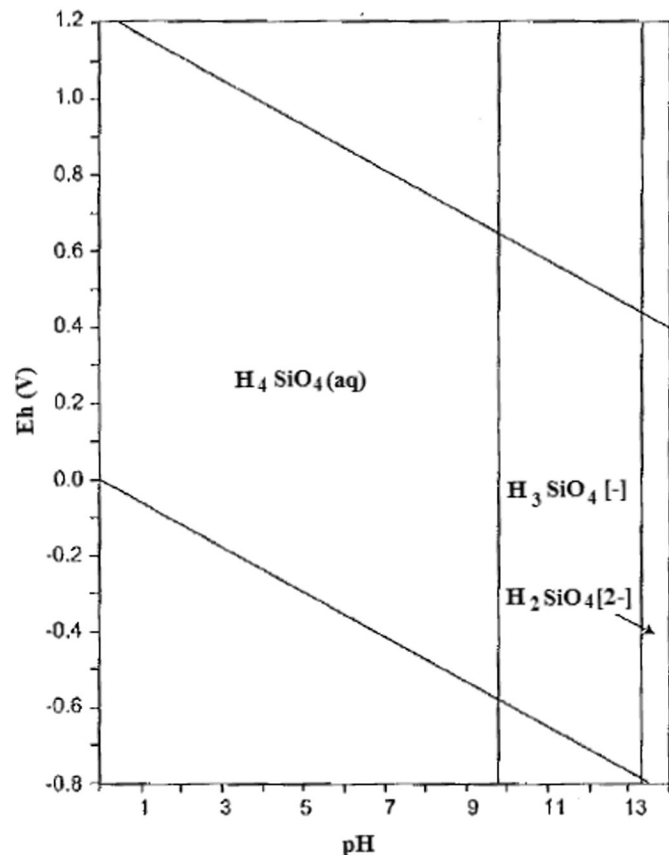


Fig. 1. Eh–pH diagram of the system Si–O–H at 298.15 K and 100 kPa (Takeno, 2005).

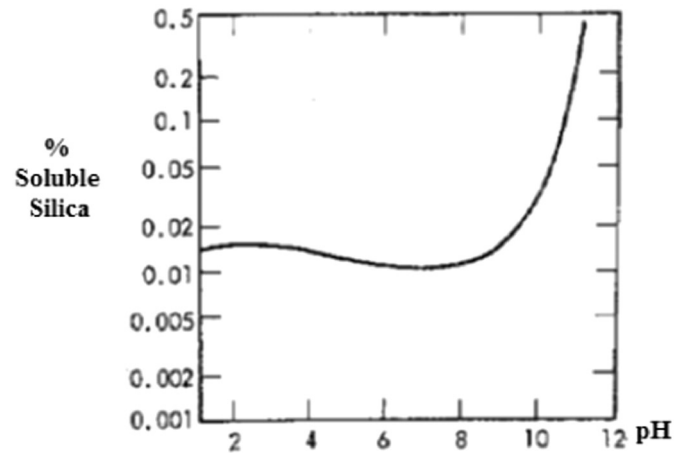


Fig. 2. Amorphous silica solubility in water at 25 °C (Queneau and Berthold, 1986).

Silica gelling is caused by the rapid polymerisation of silicic acid to form open networks. It causes dramatic increases in both plastic viscosity and yield stress that cannot be reversed by dilution. In brief, silica gel forms when silicic acid aggregates in a manner that incorporates most, if not all, of the water.

The chemistry of silica gel formation can be simply represented by:

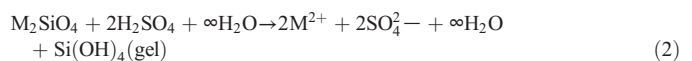


The isoelectric point of colloidal silica is pH 2. Above this point, colloidal silica is negatively charged, and below it, it is positively charged. Silica is amphoteric and always carries both charges on its surface. Below pH 2, colloidal silica grows by coalescence, which is a chaotic process and can be very fast. This growth results in a wide range of particle sizes, as well as precipitated silica and, in special cases, silica gel (Parris, 2009).

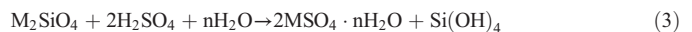
3. Quick leach chemistry (Dufresne, 1976)

The chemistry of the quick leach is based on a water-starved system and effectively rejects silica from various silicate materials.

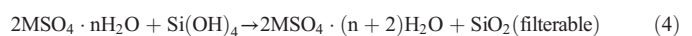
In the case of a divalent metal silicate, such as M_2SiO_4 , when attacked by sulphuric acid in the presence of excess of water, the reaction will be:



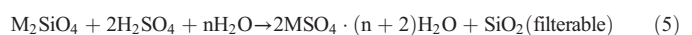
If water is restricted, the reaction will then be:



The partially hydrated metal sulphate then further reacts with silicic acid as follows:



Thus, the overall reaction is:



It is said that in a water-deficient system, the sulphate produced in the reaction indicated by Eq. (5) scavenges the available water, thus leaving little or none for the hydration of the silica for its polymerisation, and thus limiting silica polymerisation. The dehydrated silica species produced by reaction (5) is readily filterable.

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