



Effect of calcium lignosulphonate on the particulate processes during hydrogen reduction of nickel ammine sulphate solutions



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ABSTRACT

The use of additives in the precipitation of nickel with hydrogen is known to influence the particulate processes and by extension the powder properties such as morphology, microstructure and particle size distribution. Controlling these properties is crucial for some downstream processes. The present study assesses the effect of calcium lignosulphonate on the particulate processes taking place during the reduction of nickel ammine sulphate solutions by hydrogen gas. Reactions were carried out in an autoclave operated at 28 bar and 180 °C under stirring conditions of 850 rpm. Particulate processes were studied by analysing the particle size distribution and the corresponding normalized moments. These were further validated by scanning electron microscopy and nitrogen physisorption analyses. The powder phase identification and purity were determined by means of X-ray diffraction and X-ray fluorescence respectively. Calcium lignosulphonate acted as a reduction catalyst, growth promoter and by extension agglomerating agent. At 2, 5 and 7 mg/L of calcium lignosulphonate, the system was found to be dominated by breakage while agglomeration was more pronounced at 10 mg/L, as validated by scanning electron micrographs. Furthermore the use of calcium lignosulphonate resulted in the increase of the reduction rate, indicating that this additive acted as a growth promoter.

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1. Introduction

Hydrogen reduction is the most efficient and widely used method to precipitate metal powders from solutions (Meddings and Mackiw, 1965; Agrawal et al., 2006). Approximately 240,000 tons of nickel alone is produced this way per year worldwide. Commercial operations using this technology include, among others, Impala Platinum (South Africa), Sherritt (Canada) and Murrin Murrin (Australia) (Crundwell et al., 2011). Hydrogen reduction is a process whereby an ammoniacal aqueous metal salt solution is subjected to hydrogen at elevated pressures and temperatures in mechanically agitated autoclaves. Under these conditions, the dissolved metal ions undergo reduction and precipitate as metallic powders. The possibility of precipitating metals from solutions using gases as reducing agents was first established by Beketov in the 1860s (Agrawal et al., 2006). The earliest interest was in the reduction of copper sulphate solutions using sulphur dioxide and carbon monoxide as reducing agents. The use of hydrogen as a reducing agent was investigated during the period 1909–1931 in the precipitation of metals from their aqueous and organic solutions. In these investigations, reactions were done at elevated hydrogen pressure and temperature in sealed and unagitated tubes. As a result, products were contaminated with stable oxides and basic salts (Evans, 1968). The commercialization of the

hydrogen reduction process was made possible by Chemical Construction Corporation in the 1950s after an extensive research and developmental work (Habashi, 1999; Osseo-Asare, 2003).

Since the commercialization of the gaseous reduction technology, additives or addition agents have been identified as playing a major role in the operation of the process. In fact, in this process the precipitated metals have the tendency to agglomerate and plate out on the impeller of the agitator and the walls of the reaction vessel. Addition agents assist in lessening the surface activity of the reduced metal particles and therefore inhibit plating and agglomeration. Furthermore, they accelerate the reaction, making it possible to achieve reduction at shorter periods of time. They also help in controlling the physical characteristics of the obtained powder such as morphology, size and bulk density which constitute important parameters for downstream processes (Chou et al., 1976; Saarinen et al., 1998; Agrawal et al., 2006; Luidold and Antrekowitsch, 2007; Naboychenko et al., 2009). As an example, in fuel cell technologies, the porosity of materials used to construct the electrodes is a critical parameter amongst others. These elements altogether contribute in making the gaseous reduction technology commercially viable. However, commercial additives are quite expensive and in most cases their function and mechanism of action are not well understood, with most of them being employed on a trial and error basis (Bodoza et al., 2013). Since additives are normally one of the major operational costs in commercial operations, developing an understanding of their mechanism of action will enable their optimum use in industry.

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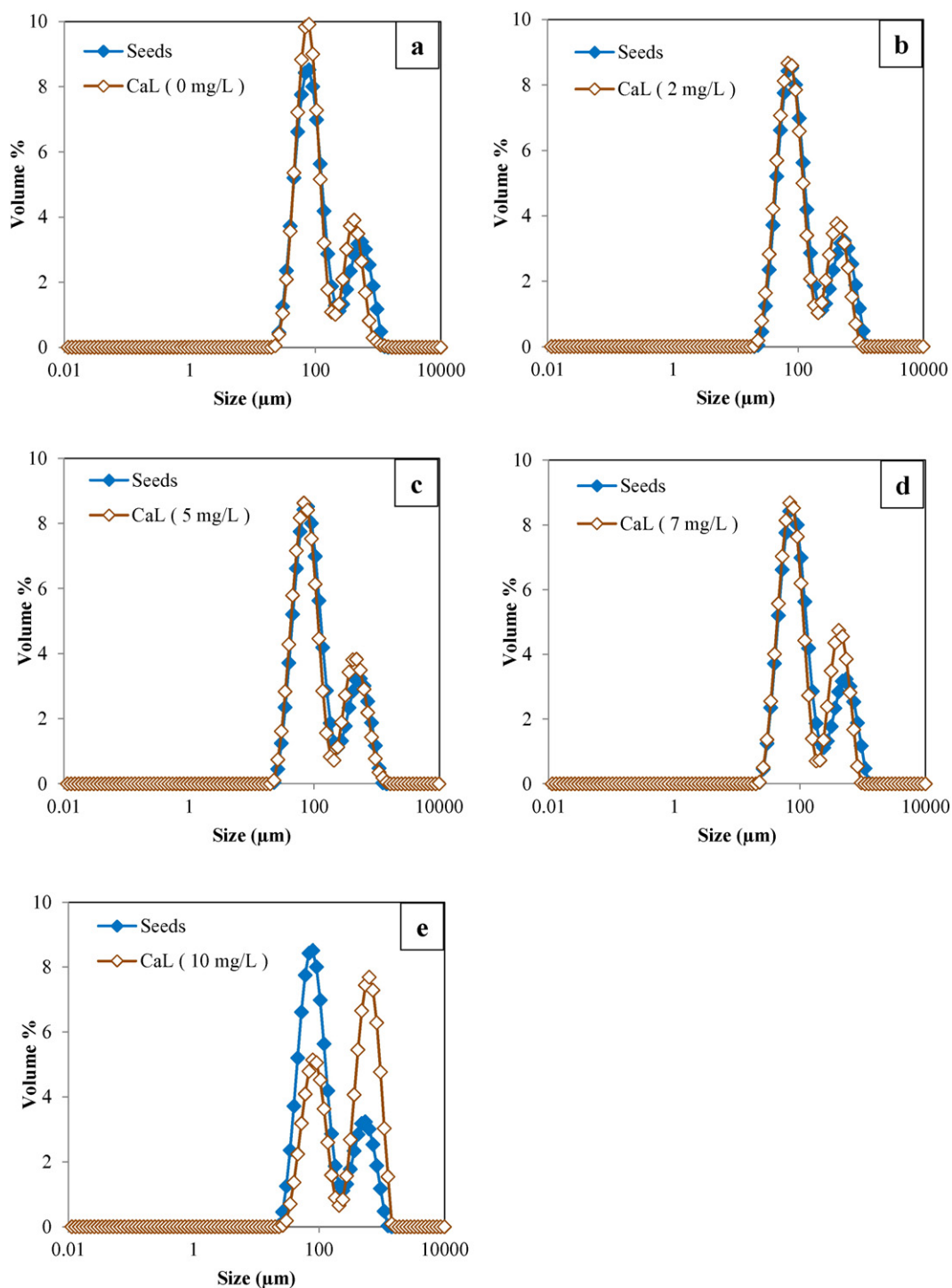


Fig. 1. Volume distributions in the absence and presence of CaL.

Table 1

Volume distribution percentiles of the seed and powder obtained at the fifth densification with and without CaL.

Powder	d(0.1) μm	d(0.5) μm	d(0.9) μm
Seed	42.399	87.488	528.436
CaL (0 mg/L)	43.133	81.482	405.324
CaL (2 mg/L)	40.161	82.431	442.220
CaL (5 mg/L)	40.269	82.654	504.417
CaL (7 mg/L)	41.501	84.521	462.440
CaL (10 mg/L)	56.901	365.261	820.288

The aim of this study was to establish the mechanisms involved in powder formation in the presence of calcium lignosulphonate in high-pressure hydrogen reduction of ammoniacal nickel sulphate solutions. This additive is of commercial importance in the production of nickel powders and has not been studied extensively. To the best of our knowledge, the only published data in literature are those of Sherritt Research and Development Division by Kunda and co-workers (Kunda et al., 1965; Kunda and Evans, 1968). In these studies, calcium lignosulphonate along with other additives were investigated at fixed concentration at the initial densification, and details on the effect of some individual additives were not reported. This paper intends to

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