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INTRODUCTION

Dear Distinguished Delegate,

The second edition of the International Conference on Advances and Innovations in Engineering (ICAIE) was held between 21-23 September 2023 at Fırat University Faculty of Engineering, Elazığ.

International Conference on Advances and Innovations in Engineering is an international scientific forum of distinguished scholars engaged in scientific, engineering and technological research, dedicated to the furtherance of science, engineering and technology. The academic research conference since its inception is at the cutting edge of international nonprofit scientific, engineering and technological progress to promoting excellence in science.

The conference plays an influential role in science and promotes developments in science, engineering and technology in a wide range of ways. The conference aims to foster research in the area of science and technology and its impact to mainstream human activities. Specifically, it serves as a venue for discussions and exchange of ideas in current issues in science and technology.

All full paper and abstract submissions to the conference are peer reviewed and refereed and evaluated based on originality, research content and correctness, relevance to contributions, and readability. In this content the full paper and abstract submissions are chosen based on technical merit, interest, applicability and how well they fit a coherent and balanced technical program. The accepted papers after rigorous peer reviewing process have been published in the refereed international conference proceedings

Chair of ICAIE Committee
Prof. Ebru AKPINAR

CONTENTS

Production of ferric-phosphate for application in the lithium battery industry

Dragana Bozic[1], Radmila Markovic[2], Ljiljana Avramovic[3], Zoran Stevanovic[4], Dejan Bugarin[5]

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Abstract

This paper presents the results of research with the aim of conquering a new technology for obtaining ferric phosphate for use in the production of lithium batteries. A lithium iron phosphate battery is a type of lithium-ion battery that has cathode materials made of lithium iron phosphate. LiFePO4 has many advantages over other lithium-ion battery designs and older lead acid (LA) batteries. They weigh less, do not require maintenance, have better charging and discharging characteristics and a much longer lifespan. Other key advantages of LiFePO4 include high amperage, longer cycle life, zero leakage and fire hazards, tolerance to sub-optimal charge and discharge cycles. Battery charging times for LiFePO4 batteries are faster than for lead acid or other lithium batteries, typically LiFePO4 units have four times the energy density and charge five times faster than lead acid batteries. The life of a LiFePO4 battery is up to five times longer than some lithium-ion batteries, often reaching up to 5000 cycles without significant performance degradation. Unlike lithium and lead-acid batteries, LiFePO4 batteries do not contain toxic, heavy or rare earth metals such as cobalt, nickel or lead. LiFePO4 consists of common materials such as graphite, iron and copper. Thus, lithium-iron-phosphate batteries are more environmentally friendly to manufacture, but also pose a much lower risk to the environment during their lifetime compared to other lithium batteries. Since the desirable characteristics of iron phosphate precursors for LFP synthesis for batteries are often different or conflict with those for other uses, the existing chemical market is an impractical source for iron phosphate precursors for LFP synthesis. In addition, iron phosphate from commercial sources synthesized by the existing method often contains impurities, for example, sulfates, chlorides, and nitrates, which are harmful to lithium-ion batteries. Moreover, different batches of commercially available iron phosphate material often have inconsistent properties. Therefore, there remains a need to develop a new technology to produce highly pure iron phosphate with consistent and desirable properties for the synthesis of LFP for batteries.

The solid residue obtained during the processing of non-standard jarosite-PbAg precipitate from the zinc production process in Elixir, Šabac was used as the starting material. In order to separate Fe from Cu, Zn, In and other useful and highly valuable components, detailed laboratory tests of the combined procedure, which includes roasting of jarosite PbAg sediment and leaching of the resulting product, were performed. After roasting a sample of Pb-Ag jarosite for 4 hours at a temperature of 5300C and leaching the resulting roasting product with water, with a phase ratio of Č:T=1:5, high leaching of the examined metals Cu (91.07%), Zn (91.97%), In (99.95%) and low leaching of Fe (9.6%) was obtained in 1 hour. These technological parameters were adopted for the tests presented in this paper. After roasting the jarosit and lying the resulting roasting product, the lye solution was separated from the solid residue by filtering. In this paper, the treatment of the solid residue to obtain ferric phosphate is presented.

Chemical composition of the solid residue was, %: Fe - 49.3; Pb - 12.0; Ag – 0.014; Cu - 0.087; Zn - 0.93. X-ray diffraction (XRD) analysis was performed on a "RigakuMiniFlex 600" instrument with a "D/teXUltra 250" high-speed detector and an X-ray tube with a copper anode. The minerals hematite (Fe_2O_3), anglesite ($PbSO_4$) and gypsum ($CaSO_4 \cdot 2H_2O$) were identified in this sample. The most common mineral is hematite, anglesite is less common, while gypsum is the least common.

Laboratory research on the leaching of the solid residue of jarosite roasting (after leaching in water) was first carried out in a sulfuric acid solution with a concentration of 10% and 65% H_2SO_4 and with the introduction of oxidants (air and ozone). Research has shown a low level of iron leaching, from 0.6 - 1%. For this reason, subsequent leaching tests were performed with hydrochloric acid of technical quality, under different conditions. The highest degree of

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Fe leaching of 91.41%, under the following conditions: ratio of phases Č:T=1:3, temperature t=800C and leaching time 1h, and represent the optimal conditions of the process of leaching in HCl. Hydroxide precipitation of Fe from alkaline solution was performed at pH=4. Alkaline solution was used for the experiments, and 2M NaOH was used as a neutralizing agent. After precipitation, the resulting precipitate was filtered and analyzed. The percentage of precipitated Fe from the ferric chloride solution was 100%.

The resulting hydroxide precipitate from alkaline solution was treated with a minimal amount of concentrated sulfuric acid until the iron precipitate was completely dissolved. The resulting ferric sulfate solution was filtered, the pH value of the filtrate was adjusted and then phosphoric acid was added.

According to literature data, using this procedure, FePO4x2H2O is obtained, while anhydrous FePO4 is obtained when drying at a temperature higher than 1800C. According to the standard (HG/T 4701-2021 China Chemistry Industry Standards), which refers to the required quality of ferric phosphate for the production of lithium batteries, the iron content in the FePO4 x2H2O product should be 28.5-30.0%. Chemical analysis of the obtained ferric phosphate showed that the obtained product was of satisfactory quality, containing 29.02% Fe. A detailed chemical analysis determined that the content of other metals (Zn, Al i Pb) in the ferric phosphate did not comply with the required standard. By recrystallization of the obtained FePO4x2H2O from the H3PO4 solution, the content of elements that deviated from the standard was reduced, whereby the following results were obtained: Pb -0.01%, Zn -0.002% and Al - 0.02%.

Keywords: Hydrometallurgy, ferric phosphate, lithium batteries, jarosite