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Technical Faculty in Bor

EcoTEK

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Ecological Truth & Environmental Research

Editor

Prof. Dr Snežana Šerbula

PROCEEDINGS

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PREFACE

The 31st international conference Ecological Truth & Environmental Research – EcoTER'24 focuses on showing the latest research findings and innovations in the field of ecology, environmental protection and sustainable development. The conference will be held in Sokobanja (Serbia) in hotel Sunce in the period of 18–21 June 2024.

The aim of the conference is to connect the experts in various fields in order to transform attitudes and behaviors in everyday practices, as well as in the industry and economy sector which is essential for achieving the desired changes that our society must undergo.

The 31st international conference Ecological Truth & Environmental Research – EcoTER'24 is organized by the University of Belgrade, Technical Faculty in Bor, and co-organized by the University of Banja Luka, Faculty of Technology; the University of Montenegro, Faculty of Metallurgy and Technology – Podgorica; the University of Zagreb, Faculty of Metallurgy – Sisak; the University of Pristina, Faculty of Technical Sciences – Kosovska Mitrovica and the Society of Young Researchers – Bor.

These Proceedings encompass 119 papers from the authors coming from the universities, research institutes and industries in 15 countries: Brazil, Norway, USA, Spain, Austria, Libya, Italy, Israel, Slovenia, Croatia, Romania, Bulgaria, Montenegro, Bosnia and Herzegovina, North Macedonia, and Serbia. It is a great honor and pleasure to cordially wish a warm welcome to all the participants of the conference.

As a part of this year's conference, the 6th Student Section – EcoTERS'24 will be held. We appreciate the contribution of the students and their mentors who have also participated in the conference and hope that students will continue to explore and to be curious, since education is a never-ending process, and knowledge is continuously growing.

The organization of the EcoTER'24 conference has been financially supported by the Ministry of Science, Technological Development and Innovation of the Republic of Serbia.

The support of the Donors and their willingness and ability to cooperate has been of great importance for the success of the EcoTER'24 conference. The organizing committee would like to extend their appreciation and gratitude to the Platinum donors of the conference – Serbia ZiJin Copper doo Bor and HBIS SERBIA, to the Gold donor of the conference – Elixir Group, as well as to the Silver donor of the conference – Serbian Chamber of Engineers.

We would like to express our sincere appreciation to all the authors who have contributed to the Proceedings. We would also like to express our gratitude to the members of the scientific, organizing and honorary committees, reviewers, speakers, chairpersons and all the conference participants for their support of the EcoTER'24. Sincere thanks go to all the people who have contributed to the successful organization of the EcoTER'24.

Prof. Snežana Šerbula,

President of the scientific and organizing committee



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APPLICATION OF THE SHRINKING CORE MODEL IN THE LEACHING PROCESS OF LiNiMnCoO_2

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Abstract

Lithium-ion batteries (LIBs) are electrochemical systems consisting of cathodes, anodes, separators and electrolytes. Improper treatment of LIBs waste can affect the quality of the environment. On the other hand, LIBs waste is a valuable source of many metals. In recent years, intensive efforts have been made to develop a hydrometallurgical recycling process for cathode materials, paying particular attention to the leaching process. The leaching of cathode material from spent LIBs is a heterogeneous reaction occurring at the solid-liquid interface. Various kinetic equations are used to process experimental results and determine the kinetic parameters of the leaching process. In this study, an overview of systems in which a shrinking core model is applied to describe the leaching process of nickel-, manganese- and cobalt-based cathode materials ($\text{LiNi}_x\text{Mn}_y\text{Co}_z\text{O}_2$) is given.

Keywords: LIBs, cathode material, kinetics, heterogeneous reactions.

INTRODUCTION

Lithium-ion batteries (LIBs) are one of the most significant sources of electrical energy due to their favorable properties such as high current density, high specific capacity, wide temperature range, low self-discharge rate, and low cost [1]. The advantage of LIBs over older generation batteries, such as lead-acid and/or nickel-cadmium batteries, lies in the lithium itself. Lithium has the lowest reduction potential of all elements (the redox potential of the Li^+/Li pair is -3.01 V), which means that lithium-based batteries have a high cell potential [2].

Commercial LIBs consist of cathodes, anodes, separators, and electrolytes. Lithium ions diffuse between the cathode and anode, facilitating the movement of electrons. The cathode materials of LIBs contain different concentrations of valuable metals depending on their structure. The most commonly used cathode materials are lithium cobalt oxide (LiCoO_2), lithium iron phosphate (LiFePO_4), lithium manganese oxide (LiMn_2O_4), and nickel, cobalt, and manganese-based cathode materials ($\text{LiNi}_x\text{Mn}_y\text{Co}_z\text{O}_2$) [3]. In recent years, nickel, cobalt, and manganese-based cathode materials (NCM) have gained increasing significance and usage in the electronics industry.

Improper handling of spent LIBs can lead to contamination of soil, water, and air. On the other hand, spent LIBs represent valuable waste material for the recovery of valuable metals such as lithium, cobalt, nickel, manganese, and/or their compounds [4].

The recycling technologies used for the processing of cathode materials from spent LIBs can be divided into pyrometallurgical, hydrometallurgical and bio-hydrometallurgical processes [5–7]. Pyrometallurgical processes release toxic gases and a large amount of energy, and the percentage of material loss is high because the process is carried out at temperatures above 1400°C [8]. Biohydrometallurgical processes require a considerable amount of time and the presence of microorganisms for efficient incubation [9]. The application of hydrometallurgical treatment requires high metal purity, minimal gas emissions and low energy consumption. The most important step in the entire hydrometallurgical process is acid leaching [4].

During the hydrometallurgical process, especially in the leaching of metals from cathode material, it is necessary to understand the kinetics of heterogeneous chemical reactions, which enables the identification and definition of the appropriate reaction mechanism. The most commonly used kinetic models to represent heterogeneous chemical reactions during the metal leaching process from cathode material are the shrinking core model, the logarithmic model and the Avrami model [10,11].

This paper provides an overview of leaching systems in which the shrinking core model was used to determine the kinetic parameters of the cathode material leaching process.

KINETICS AND MECHANISM OF LEACHING NICKEL, COBALT, AND MANGANESE-BASED CATHODE MATERIAL

The kinetic equations used to describe the leaching process of cathode material can be classified into four models: a model controlled by a chemical reaction (reaction 1), a model controlled by a diffusion process (reaction 2), the logarithmic model (reaction 3), and the Avrami model (reaction 4) [11].

$$1-(1-x)^{1/3} = k_1 t \quad (1)$$

$$1 - 2/3x - (1-x)^{2/3} = k_2 t \quad (2)$$

$$(-\ln(1-x))^2 = k_3 t \quad (3)$$

$$-\ln(1-x) = k_4 t^n \quad (4)$$

where: x - conversion degree; t - leaching time (min); k_1 , k_2 , k_3 , and k_4 - rate constants (1–4) (min^{-1}).

In order to determine the control level of the process rate, the experimental data must be adapted to the existing kinetic equations. A high value for the correlation coefficient (R^2) indicates the level that determines the reaction rate in the process under consideration. The value of the activation energy is calculated using the Arrhenius equation.

Numerous factors affect the kinetics of heterogeneous chemical reactions during the decomposition of cathode material, in particular the concentration of the leaching agent,

concentration of the reducing agent, temperature, time, and solid-liquid ratio. In addition, the pretreatment of the cathode material and the state of battery decomposition can significantly influence the metal leaching rate and activation energy values [12].

Table 1 contains literature data on the decomposition system of nickel, cobalt and manganese-based cathode materials as well as the kinetic equations used to determine the activation energy values. The data listed in Table 1 show that the shrinking core model is generally used to describe the kinetics and mechanism of leaching of LiNiMnCoO₂.

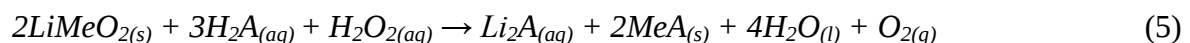
Table 1 Kinetic equations for the leaching process of cathode material

Cathode Material	Leaching System	Kinetic equation	Activation energy (kJ/mol)	References
LiNi _x Co _x Mn _x O ₂	C ₄ H ₆ O ₆ + H ₂ SO ₄ + H ₂ O ₂	$1 - (1 - x)^{1/3} = kt$	Li:49.7 Ni:48.5 Co:50.0 Mn:47.6	[12]
LiNi _x Co _x Mn _x O ₂	NH ₃ + (NH ₄) ₂ SO ₄ + NaSO ₃	$1 - (1 - x)^{1/3} = kt$	Li:62.0 Ni:76.3 Co:81.5	[11]
LiNi _x Co _x Mn _x O ₂	subcritical water + PVDF	$1 - (1 - x)^{1/3} = kt$	Li:63.7 Ni:73.5 Co:74.9 Mn:71.8	[13]
LiNi _x Co _x Mn _x O ₂	H ₂ SO ₄ + H ₂ S	$1 - 2/3x - (1 - x)^{2/3} = kt$	Li:23.3 Ni:28.7 Co:27.6 Mn:26.0	[14]
LiNi _x Co _x Mn _x O ₂	H ₂ SO ₄ + H ₂ O ₂	$1 - 2/3x - (1 - x)^{2/3} = kt$	Li:20.1 Ni:34.3 Co:29.9 Mn:20.8	[15]
LiNi _x Co _x Mn _x O ₂	C ₂ H ₂ O ₄ + H ₂ O ₂	$(-\ln(1 - x))^2 = k_3t$	Li:9.7 Ni:11.2 Co:9.5 Mn:6.2	[16]
LiNi _x Co _x Mn _x O ₂	H ₂ SO ₄ + ginkgo biloba	$1 - (1 - x)^{1/3} = kt$ $1 - 2/3x - (1 - x)^{2/3} = kt$	Li:23.4 Ni:74.6 Co:79.3 Mn:73.1	[17]

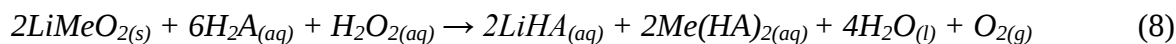
SHRINKING CORE MODEL

Chen *et al.* [12] used the shrinking core model to investigate the dissolution kinetics of LiNi_xMn_yCo_zO₂ using sulfuric acid and tartaric acid and with the addition of hydrogen peroxide as a reducing agent.

The leaching mechanism of LiNi_xMn_yCo_zO₂ with tartaric acid can be illustrated by the reactions (5–8). If the ratio of cathode material (LiMeO₂, where Me represents Ni, Co, and Mn) to tartaric acid (H₂A) is 2:3, the soluble complex Li₂A and the insoluble complexes MeA are formed according to reaction 5.



The soluble complex Li_2A and the insoluble complexes MeA can be dissolved by adding tartaric acid so that the ratio of cathode material to tartaric acid is 1:3.

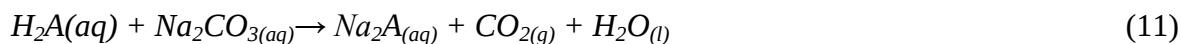
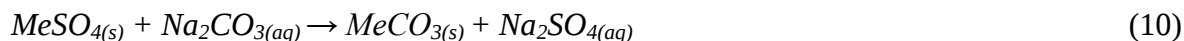


In the leaching of cathode material with tartaric acid, hydrogen peroxide is used to convert cobalt and manganese from higher oxidation states (Co^{3+} i Mn^{4+}) to lower oxidation states (Co^{2+} i $\text{Mn}^{3+}/\text{Mn}^{2+}$).

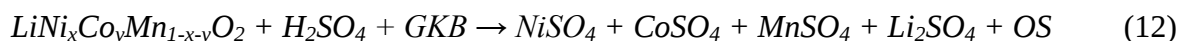
When $\text{LiNi}_x\text{Mn}_y\text{Co}_z\text{O}_2$ is leached with tartaric acid, complexes are formed from which valuable metals cannot be extracted directly due to the disturbed crystal structure. However, the tartaric acid complexes can be easily dissolved by adding sulfuric acid to the leaching solution, whereby transition metal sulphates (MeSO_4) are formed after the following reaction:



In order to ensure complete precipitation of the Me^{2+} ions, sodium carbonate is added to the resulting sulphates, resulting in transition metal carbonates:



Zhu *et al.* [17] used the shrinking core model to study the leaching kinetics of $\text{LiNi}_x\text{Co}_y\text{Mn}_{1-x-y}\text{O}_2$ in the H_2SO_4 + Ginkgo biloba system. The leaching mechanism of $\text{LiNi}_x\text{Co}_y\text{Mn}_{1-x-y}\text{O}_2$ cathode material, under the influence of sulfuric acid and Ginkgo biloba can be represented by the following reaction:



where: GKB - Ginkgo biloba, reducing agent; OS - oxidation product.

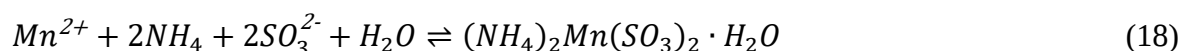
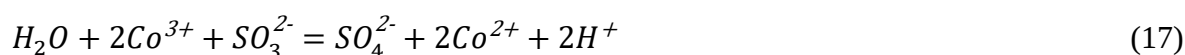
The dissolution mechanism of $\text{LiNi}_x\text{Co}_y\text{Mn}_{1-x-y}\text{O}_2$ cathode material under the influence of sulfuric acid and Ginkgo biloba consists of three main stages [17]:

- Diffusion of sulfuric acid and Ginkgo biloba molecules to the surface of the cathode material by liquid flow,
- Penetration and diffusion of sulfuric acid and Ginkgo biloba molecules through the product layer to the surface of the unreacted core, and
- Chemical reaction of sulfuric acid and Ginkgo biloba with $\text{LiNi}_x\text{Co}_y\text{Mn}_{1-x-y}\text{O}_2$ at the reaction surface.

Wang *et al.* [11] determined the kinetics and mechanism of leaching of transition metals from $\text{LiNi}_x\text{Co}_x\text{Mn}_x\text{O}_2$ using the shrinking core model in the $\text{NH}_3\text{-(NH}_4)_2\text{SO}_4\text{-Na}_2\text{SO}_3$ system. During the leaching process, Ni^{2+} , Co^{2+} , and Mn^{2+} ions react with free ammonia to form stable amino complexes, corresponding to the following reactions:



Ammonium sulfate sulphate is used to facilitate the transition of cobalt from a higher oxidation state to a lower oxidation state. At temperatures above 70°C , the reduction of ammonium sulfate to ammonium sulfite takes place, which facilitates the easier separation of manganese compounds [11].



In addition to the formation of amino complexes, sulfates, carbonates, and oxides can also be formed, e.g. Li_2CO_3 , CoSO_4 , NiSO_4 , and/or Co_3O_4 . Manganese-amino complexes are unstable, and Mn^{2+} ions can react with CO_3^{2-} ions to form stable manganese carbonate complexes, according to the following reaction:



At operating temperatures above 70°C , manganese ions change from the Mn^{4+} oxidation state to the Mn^{2+} oxidation state and form unstable complexes, clathrates, manganese-ammonium-sulfite hydrates, $(\text{NH}_4)_2\text{Mn}(\text{SO}_3)_2 \cdot \text{H}_2\text{O}$, which leads to reduced manganese leaching [11].

Nshizirungu *et al.* [13] investigated the dissolution mechanism of $\text{LiNi}_x\text{Co}_x\text{Mn}_x\text{O}_2$ in a polyvinylidene fluoride (PVDF) solution. Based on the shrinking core model, the authors assumed that the leaching of the transition metals occurs in two stages. In the first stage, the decomposition of PVDF takes place in the aqueous solution in the temperature range of 270°C – 350°C . In the second stage, with an increase in the temperature range (350°C – 370°C), secondary reactions take place between fluoride and hydroxyl ions. As a result, transition metal hydroxides and hydrofluoric acid are formed according to the following reactions:





CONCLUSION

Spent lithium-ion batteries are a valuable waste material for the recovery of lithium, cobalt, nickel, manganese, and/or their compounds. However, if not handled properly, this type of electronic waste can pose a risk to the environment and human health. However, if not treated properly, this type of electronic waste can endanger the environment and human health. Numerous studies conducted to better understand the systems involved in the recycling process of cathode material show that the shrinking core model is often used to describe the leaching process. Various systems of organic and inorganic origin and their combinations are used as leaching agents. The calculated activation energy values for Li, Ni, Mn, and Co indicate that for most systems the process is determined by the chemical reaction between the leaching agent and the unreacted particles of LiNiMnCoO₂.

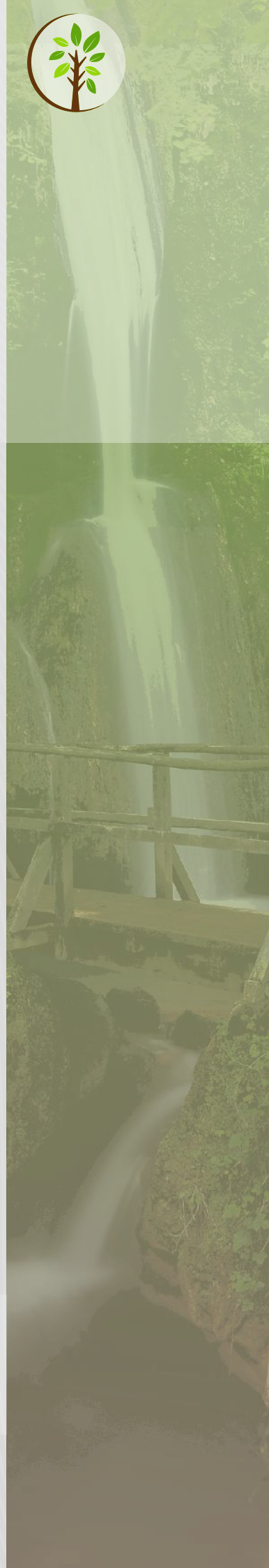
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