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Non-isothermal reaction mechanism and kinetic analysis for the synthesis of monoclinic lithium zirconate (m-Li_2ZrO_3) during solid-state reaction

Juan P. Yasnó[1*], Susana Conconi[1,2], Arnaldo Visintin[2,3] and Gustavo Suárez[1,2]

Abstract

Non-isothermal reaction mechanism and kinetic analysis for the synthesis of monoclinic lithium zirconate (m-Li_2ZrO_3) were investigated by processing of TG-DTA, along with XRD, DLS, and HRTEM. For this purpose, the solid-state reaction of Li_2CO_3 with ZrO_2 was carried out by TG-DTA at different heating rates (10, 20, and 30 °C/min) from room temperature to 1100 °C. The thermal data was used to calculate the kinetic parameters by two types of isoconversional methods: Flynn-Wall-Ozawa (FWO) and Kissinger-Akahira-Sunose (KAS). The reaction mechanism was determined by the model-fitting method, applying the Coats-Redfern (CR) approximation to the different solid-state reaction models. The results confirmed the formation of pure m-Li_2ZrO_3, consists of semispherical particles of about 490 nm, using a very short reaction time. The average activation energy obtained by FWO and KAS methods were 274.73 and 272.50 kJ/mol, respectively. It was found that the formation of m-Li_2ZrO_3 from Li_2CO_3 with ZrO_2 is governed by the three-dimensional diffusion mechanism. Based on these results, a microscopic reaction model of the formation of m-Li_2ZrO_3 was proposed.

Keywords: M-Li_2ZrO_3, Solid-state reaction kinetics, Non-isothermal, TG-DTA, XRD

Introduction

Monoclinic lithium zirconate (m-Li_2ZrO_3) is a ceramic material that has potential applications in different fields including solid-state lithium-ion batteries (Doeng et al. 2015; Sherstobitova et al. 2016; Zhan et al. 2018), solid sorbent for CO_2 capture (Ida and Lin 2003; Kordatos et al. 2017; Chattaraj 2017), and nuclear reactors (Taddia et al. 2005; Oyaidzu et al. 2006; Chitnis et al. 2018). While m-Li_2ZrO_3 is conventionally prepared via solid-state reaction of Li_2CO_3 with ZrO_2 (see Eq.(1)) (Wyers and Cordfunke 1989; Pfeiffer and Knowles 2004; Sree Rama Murthy et al. 2017), its formation kinetics has not been reported in the scientific literature. In the bibliographic review on reaction kinetics, a study was found on the decomposition of m-Li_2ZrO_3 into ZrO_2 (Pfeiffer and Knowles 2004). It is worth highlighting the fact that the knowledge of the reaction kinetics and mechanism are very important in order to optimize the solid-state process for large-scale production and advanced applications. Moreover, reasonable mechanistic conclusions can be drawn from the kinetic data (Lu and Wu 2008; Mandal 2014).

$$Li_2CO_3 + ZrO_2 \xrightarrow{\Delta} m - Li_2ZrO_3 + CO_2\uparrow \quad (1)$$

Some of the analytical methods used to study the reaction kinetics in the solid-state are thermogravimetric analysis (TGA), differential-thermal analysis (DTA), X-ray diffraction (XRD), differential-scanning calorimetry (DSC), and nuclear magnetic resonance (NMR) (Mandal 2014; Vyazovkin et al. 2011; Khawam and Flanagan

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Non Isothermal Reaction Analysis

Dietrich H. Welte,Brian Horsfield,Donald R. Baker

Non Isothermal Reaction Analysis:

Non-isothermal Reaction Analysis Erhard Koch,1977 **The Investigation of Organic Reactions and Their Mechanisms** Howard Maskill,2008-04-15 A range of alternative mechanisms can usually be postulated for most organic chemical reactions and identification of the most likely requires detailed investigation Investigation of Organic Reactions and their Mechanisms will serve as a guide for the trained chemist who needs to characterise an organic chemical reaction and investigate its mechanism but who is not an expert in physical organic chemistry Such an investigation will lead to an understanding of which bonds are broken which are made and the order in which these processes happen This information and knowledge of the associated kinetic and thermodynamic parameters are central to the development of safe efficient and profitable industrial chemical processes and to extending the synthetic utility of new chemical reactions in chemical and pharmaceutical manufacturing and academic environments Written as a coherent account of the principal methods currently used in mechanistic investigations at a level accessible to academic researchers and graduate chemists in industry the book is highly practical in approach The contributing authors an international group of expert practitioners of the techniques covered illustrate their contributions by examples from their own research and from the relevant wider chemical literature The book covers basic aspects such as product analysis kinetics catalysis and investigation of reactive intermediates It also includes material on significant recent developments e g computational chemistry calorimetry and electrochemistry in addition to topics of high current industrial relevance e g reactions in multiphase systems and synthetically useful reactions involving free radicals and catalysis by organometallic compounds Reactions and Mechanisms in Thermal Analysis of Advanced Materials Atul Tiwari,Baldev Raj,2015-08-06 Strong bonds form stronger materials For this reason the investigation on thermal degradation of materials is a significantly important area in research and development activities The analysis of thermal stability can be used to assess the behavior of materials in the aggressive environmental conditions which in turn provides valuable information about the service life span of the materiel Unlike other books published so far that have focused on either the fundamentals of thermal analysis or the degradation pattern of the materials this book is specifically on the mechanism of degradation of materials The mechanism of rapturing of chemical bonds as a result of exposure to high temperature environment is difficult to study and resulting mechanistic pathway hard to establish Limited information is available on this subject in the published literatures and difficult to excavate Chapters in this book are contributed by the experts working on thermal degradation and analysis of the wide variety of advanced and traditional materials Each chapter discusses the material its possible application behavior of chemical entities when exposed to high temperature environment and mode and the mechanistic route of its decomposition Such information is crucial while selecting the chemical ingredients during the synthesis or development of new materials technology *Introduction to Chemical Reactor Analysis* R.E. Hayes,2020-12-17 This book provides an introduction to the basic concepts of chemical reactor analysis and design It is

intended for both the senior level undergraduate student in chemical engineering and the working professional who may require an understanding of the basics of this subject Handbook of Thermal Analysis and Calorimetry Michael E. Brown,1998-09-07 Handbook of Thermal Analysis and Calorimetry Volume 1 Principles and Practice describes the basic background information common to thermal analysis and calorimetry in general Thermodynamic and kinetic principles are discussed along with the instrumentation and methodology associated with thermoanalytical and calorimetric techniques The purpose is to collect the discussion of these general principles and minimize redundancies in the subsequent volumes that are concerned with the applications of these principles and methods More unique methods which pertain to specific processes or materials are covered in later volumes *Handbook of Thermal Analysis and Calorimetry* ,2018-03-12 Handbook of Thermal Analysis and Calorimetry Recent Advances Techniques and Applications Volume Six Second Edition presents the latest in a series that has been well received by the thermal analysis and calorimetry community This volume covers recent advances in techniques and applications that complement the earlier volumes There has been tremendous progress in the field in recent years and this book puts together the most high impact topics selected for their popularity by new editors Sergey Vyazovkin Nobuyoshi Koga and Christoph Schick all editors of Thermochimica Acta Among the important new techniques covered are biomass conversion sustainable polymers polymer nanocompsoties nonmetallic glasses phase change materials propellants and explosives applications to pharmaceuticals processes in ceramics metals and alloys ionic liquids fast scanning calorimetry and more Features 19 all new chapters to bring readers up to date on the current status of the field Provides a broad overview of recent progress in the most popular techniques and applications Includes chapters authored by a recognized leader in each field and compiled by a new team of editors each with at least 20 years of experience in the field of thermal analysis and calorimetry Enables applications across a wide range of modern materials including polymers metals alloys ceramics energetics and pharmaceutics Overviews the current status of the field and summarizes recent progress in the most popular techniques and applications A Thermo-Economic Approach to Energy from Waste Anand Ramanathan,Meera Sheriffa Begum,Amaro Olimpio Pereira,Claude Cohen,2021-10-26 A Thermo Economic Approach to Energy From Waste provides readers with the tools to analyze the effectiveness of biomass waste conversion into value added products and how thermochemical conversion methods can be commercialized with minimum environmental impact The book provides a comprehensive overview of biomass conversion technologies through pyrolysis including the types of reactors available reactor mechanisms and the upgradation of bio oil Case studies are provided on waste disposal in selected favelas slums of Rio de Janeiro including data on subnormal clusters and analyses of solid waste in the 37 slums of Catumbi Step by step guidance is provided on how to use a life cycle assessment LCA approach to analyze the potential impact of various waste to energy conversion technologies and a brief overview of the common applications of LCA in other geographical locations is presented including United States Europe China and Brazil Finally waste to value added functional

catalysts for the transesterification process in biodiesel production are discussed alongside various other novel technologies for biodiesel production process simulation and techno economic analysis of biodiesel production Bringing together research and real world case studies from an LCA perspective the book provides an ideal reference for researchers and practitioners interested in waste to energy conversion LCA and the sustainable production of bioenergy Presents an overview of the technologies for the production of biofuels from waste via pyrolysis and gasification Provides a guide to the utilization of LCA to assess the economic and environmental impact of value added products Describes real world case studies on the implementation of LCA in waste to energy scenarios *Applied Mechanics Reviews* ,1948 *Reactions in the Solid State* Michael E. Brown,D. Dollimore,A.K. Galwey,1980-01-01 The whole of Volume 22 is devoted to the kinetics and mechanisms of the decomposition and interaction of inorganic solids extended to include metal carboxylates After an introductory chapter on the characteristic features of reactions in the solid phase experimental methods of investigation of solid reactions and the measurement of reaction rates are reviewed in Chapter 2 and the theory of solid state kinetics in Chapter 3 The reactions of single substances loosely grouped on the basis of a common anion since it is this constituent which most frequently undergoes breakdown are discussed in Chapter 4 the sequence being effectively that of increasing anion complexity Chapter 5 covers reactions between solids and includes catalytic processes where one solid component remains unchanged double compound formation and rate processes involving the interactions of more than three crystalline phases The final chapter summarises the general conclusions drawn in the text of Chapter 2 5 *Poly(lactic acid)* Rafael A. Auras,Loong-Tak Lim,Susan E. M. Selke,Hideto Tsuji,2022-06-01 POLY LACTIC ACID The second edition of a key reference fully updated to reflect new research and applications Poly lactic acid s PLAs biodegradable polymers derived from lactic acid have become vital components of a sustainable society Eco friendly PLA polymers are used in numerous industrial applications ranging from packaging to medical implants and to wastewater treatment The global PLA market is predicted to expand significantly over the next decade due to increasing demand for compostable and recyclable materials produced from renewable resources Poly lactic acid Synthesis Structures Properties Processing Applications and End of Life provides comprehensive coverage of the basic chemistry production and industrial use of PLA Contributions from an international panel of experts review specific processing methods characterization techniques and various applications in medicine textiles packaging and environmental engineering Now in its second edition this fully up to date volume features new and revised chapters on 3D printing the mechanical and chemical recycling of PLA PLA stereocomplex crystals PLA composites the environmental footprint of PLA and more Highlights the biodegradability recycling and sustainability benefits of PLA Describes processing and conversion technologies for PLA such as injection molding extrusion blending and thermoforming Covers various aspects of lactic acid lactide monomers including physicochemical properties and production Examines different condensation reactions and modification strategies for enhanced polymerization of PLA Discusses the thermal rheological and mechanical

properties of PLA Addresses degradation and environmental issues of PLA including photodegradation radiolysis hydrolytic degradation biodegradation and life cycle assessment Poly lactic acid Synthesis Structures Properties Processing Applications and End of Life Second Edition remains essential reading for polymer engineers materials scientists polymer chemists chemical engineers industry professionals using PLA and scientists and advanced student engineers interested in biodegradable plastics **Tenth International Symposium on Chemical Reaction Engineering** J. R. Bourne,W. Regenass,W. Richarz,2017-05-04 ISCRE 10 Tenth International Symposium on Chemical Reaction Engineering documents the proceedings of the symposium which brought together experts from all over the world to discuss developments in CRE Efforts were made to cover high added value substances and to encourage papers from industry Some success was achieved but there remain significant gaps between Chemists and Chemical Engineers when considering high added value products as well as between researchers and practitioners of CRE The volume begins with plenary papers covering topics such as challenges in reactor modeling bioreactor engineering the design of reaction systems for specialty organic chemicals This is followed by papers presented during the eight technical sessions Technical session A focused on the modeling and control of chemical reactions Technical session B was devoted to studies on biotechnology Technical session C covered mixing while Technical session D dealt with special reactor systems and chemicals The papers in Technical session E examined reactions for emission control and recycling Technical session F covered the safety aspects of CRE Technical session G focused on the experiments with multiphase reactions while Technical session H dealt with catalytic reactors Solid Waste Management Garima Chauhan,Surajbhan Sevda,2023-12-01 Waste is generally identified as goods or material that are perceived to be mostly valueless However objects that are perceived to be waste based on consumers object valuation can be redefined to create value This requires a multitude of efforts using different strategies in waste prevention and management This book is an edited collection of various chemical approaches used for valorization of solid wastes particularly waste electrical and electronic equipment plastic waste and agro residue waste that provide research insights into the concept waste to energy Covering a variety of interdisciplinary topics on waste treatment and resource recovery makes the book one for all that serves as an excellent reading material for engineers science scholars entrepreneurs and organizations who are working in the field of waste management *Bioprocess Engineering* Shijie Liu,2012-11-21 Bioprocess Engineering involves the design and development of equipment and processes for the manufacturing of products such as food feed pharmaceuticals nutraceuticals chemicals and polymers and paper from biological materials It also deals with studying various biotechnological processes Bioprocess Kinetics and Systems Engineering first of its kind contains systematic and comprehensive content on bioprocess kinetics bioprocess systems sustainability and reaction engineering Dr Shijie Liu reviews the relevant fundamentals of chemical kinetics including batch and continuous reactors biochemistry microbiology molecular biology reaction engineering and bioprocess systems engineering introducing key principles that enable

bioprocess engineers to engage in the analysis optimization design and consistent control over biological and chemical transformations The quantitative treatment of bioprocesses is the central theme of this book while more advanced techniques and applications are covered with some depth Many theoretical derivations and simplifications are used to demonstrate how empirical kinetic models are applicable to complicated bioprocess systems Contains extensive illustrative drawings which make the understanding of the subject easy Contains worked examples of the various process parameters their significance and their specific practical use Provides the theory of bioprocess kinetics from simple concepts to complex metabolic pathways Incorporates sustainability concepts into the various bioprocesses **1991 International Conference on Coal Science Proceedings** Sam Stuart,2013-09-17 1991 International Conference on Coal Science Proceedings **Treatise on Process Metallurgy** Alexander McLean,Roderick Guthrie,Sridhar Seetharaman,H. Y. Sohn,2025-06-16 Treatise on Process Metallurgy Volume Two Process Phenomena provides academics with the fundamentals of the manufacturing of metallic materials from raw materials into finished parts or products In these fully updated volumes coverage is expanded into four volumes including Process Fundamentals encompassing process fundamentals structure and properties of matter thermodynamic aspects of process metallurgy and rate phenomena in process metallurgy Processing Phenomena encompassing interfacial phenomena in high temperature metallurgy metallurgical process phenomena and metallurgical process technology Metallurgical Processes encompassing mineral processing aqueous processing electrochemical material and energy processes and iron and steel technology non ferrous process principles and production technologies and more The work distills the combined academic experience from the principal editor and the multidisciplinary four member editorial board Provides the entire breadth of process metallurgy in a single work Includes in depth knowledge in all key areas of process metallurgy Approaches the topic from an interdisciplinary perspective providing broad range coverage on topics **Laser Induced Damage in Optical Materials, 1986** Harold Earl Bennett,1988 **Petroleum and Basin Evolution** Dietrich H. Welte,Brian Horsfield,Donald R. Baker,2012-12-06 This book has been prepared by the collaborative effort of two somewhat separate technical groups the researchers at the Institute for Petroleum and Organic Geochemistry Forschungszentrum Jii lich KFA and the technical staff of Integrated Exploration Systems IES One of us Donald R Baker from Rice University Houston has spent so much time at KFA as a guest scientist and researcher that it is most appropriate for him to contribute to the book During its more than 20 year history the KFA group has made numerous and significant contributions to the understanding of petroleum evolution The KFA researchers have emphasized both the field and laboratory approaches to such important problems as source rock recognition and evaluation oil and gas generation maturation of organic matter expulsion and migration of hydrocarbons and crude oil composition and alteration IES Jiilich has been a leader in the development and application of numerical simulation basin modeling procedures The cooperation between the two groups has resulted in a very fruitful synergy effect both in the development of modeling software and in its

application The purpose of the present volume developed out of the 1994 publication by the American Association of Petroleum Geologists of a collection of individually authored papers entitled The Petroleum System From Source to Trap edited by L B Magoon and W G Dow **Handbook of Benzoxazine Resins** Hatsuo Ishida,Tarek Agag,2011-07-13 This handbook provides a wide overview of the field fundamental understanding of the synthetic methods and structure property correlation as well as studies related to applications in a wide range of subjects The handbook also provides 1H and 13C NMR spectra FTIR spectra DSC and TGA thermograms to aid in research activities Additional tables on key NMR and FTIR frequencies unique to benzoxazine heat of polymerization Tg and char yield will greatly aid in the choice of proper benzoxazine for a specific application Provides thorough coverage of the chemistry and applications of benzoxazine resins with an evidence based approach to enable chemists engineers and material scientists to evaluate effectiveness Features spectra which allow researchers to compare results avoid repetition and save time as well as tables on key NMR frequency IR frequency heat of polymerization of many benzoxazine resins to aid them in selection of materials Written by the foremost experts in the field *Dispersive Kinetics* Andrzej Plonka,2013-04-17 Dynamical processes in which many timescales coexist are called dispersive The rate coefficients for dispersive processes depend on time In the case of a chemical reaction the time dependence of the rate coefficient k t termed the specific reaction rate is rationalized in the following way Reactions by their very nature have to disturb reactivity distributions of the reactants in condensed media as the more reactive species are the first ones to disappear from the system The extent of this disturbance depends on the ratio of the rates of reactions to the rate of internal rearrangements mixing in the system restoring the initial distribution in reactivity of reactants If the rates of chemical reactions exceed the rates of internal rearrangements then the initial distributions in reactant reactivity are not preserved during the course of reactions and the specific reaction rates depend on time Otherwise the extent of disturbance is negligible and classical kinetics with a constant specific reaction rate k termed the reaction rate constant may be valid as an approximation In condensed media dispersive dynamical processes are endemic and this is the first monograph devoted to these processes **Energy Research Abstracts** ,1982

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