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Non-isothermal reaction mechanism and kinetic analysis for the synthesis of monoclinic lithium zirconate (m-Li₂ZrO₃) during solid-state reaction

Juan P. Yasno^{1*}, Susana Conconi^{1,2}, Arnaldo Vainšin^{2,3} and Gustavo Suárez^{3,2}

Abstract

Non-isothermal reaction mechanism and kinetic analysis for the synthesis of monoclinic lithium zirconate (m-Li₂ZrO₃) were investigated by processing of TG-DTA, along with XRD, DLS, and HRTEM. For this purpose, the solid-state reaction of Li₂CO₃ with ZrO₂ 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-Li₂ZrO₃, 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-Li₂ZrO₃ from Li₂CO₃ with ZrO₂ is governed by the three-dimensional diffusion mechanism. Based on these results, a microscopic reaction model of the formation of m-Li₂ZrO₃ was proposed.

Keywords: M-Li₂ZrO₃, Solid-state reaction kinetics, Non-isothermal, TG-DTA, XRD

Introduction

Monoclinic lithium zirconate (m-Li₂ZrO₃) is a ceramic material that has potential applications in different fields including solid-state lithium-ion batteries (Dong et al. 2015; Sherstobitova et al. 2016; Zhan et al. 2018), solid sorbent for CO₂ capture (Ida and Lin 2003; Kordatos et al. 2017; Chattaraj 2017), and nuclear reactors (Taddia et al. 2005; Oyaizxa et al. 2006; Chitnis et al. 2018). While m-Li₂ZrO₃ is conventionally prepared via solid-state reaction of Li₂CO₃ with ZrO₂ (see Eq.(1)) (Wyers and Cordfunke 1989; Pfeiffer and Knowles 2004; See 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-Li₂ZrO₃ into ZrO₂ (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).



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

* Correspondence: jpyasno@cetmrc.unipue.edu.ar; jpyasno@hotmail.com

¹CETMRC Technological Center of Mineral Resources and Ceramics (UNLP-CIC-CONICET), Cno. Centenario y 506, M.B. Gonnet, 1897 Buenos Aires, Argentina

Full list of author information is available at the end of the article

Non Isothermal Reaction Analysis

J. I. Owens



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 material 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 rupturing 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

[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 nanocomposites 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 pharmaceuticals 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 **Thermal Analysis Kinetics for Understanding Materials**

Behavior Sergey Vyazovkin, 2020-12-29 Changing the temperature of a substance can stimulate dramatic changes of its state These changes can be intermolecular physical and intramolecular chemical in nature Physical changes occur without breaking intramolecular bonds and lead to transitions between the four major phases gas liquid crystal and glass Chemical changes are associated with chemical reactions that originate from breaking intramolecular bonds Phase transitions as well as chemical reactions occur at finite rates Measuring the rates of processes is the realm of kinetics The kinetics of thermally stimulated processes is routinely measured using thermal analysis techniques such as differential scanning calorimetry DSC and thermogravimetric analysis TGA Knowing the process rates and their dependence on temperature is of vital importance for understanding the behavior of materials exposed to variations in temperature In recent years thermal analysis kinetics has made significant progress by developing computational tools for reliable kinetic analysis It has also expanded its traditional application area to newly developed nano and biomaterials This Special Issue is a series of papers that reflect recent developments in the field and highlight the essential role of thermal analysis kinetics in understanding the processes responsible for the thermal behavior of various materials **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 **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 Jülich 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 Jilich 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 ^{13}C 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 T_g 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

Metal-water Reactions J. I. Owens,1959 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

Springer Handbook of Advanced Catalyst Characterization Israel E. Wachs,Miguel A. Bañares,2023-05-17 Co edited by world renowned scientists in the field of catalysis this book contains the cutting edge in situ and operando spectroscopy characterization techniques operating under reaction conditions to determine a materials bulk surface and solution complex

and their applications in the field of catalysis with emphasis on solid catalysts in powder form since such catalyst are relevant for industrial applications The handbook covers from widely used to cutting edge techniques The handbook is written for a broad audience of students and professionals who want to pursue the full capabilities available by the current state of the art in characterization to fully understand how their catalysts really operate and guide the rational design of advanced catalysts Individuals involved in catalysis research will be interested in this handbook because it contains a catalogue of cutting edge methods employed in characterization of catalysts These techniques find wide use in applications such as petroleum refining chemical manufacture natural gas conversion pollution control transportation power generation pharmaceuticals and food processing

Rheology and Processing of Polymeric Materials Chang Dae Han, 2007-05-16 Volume 1 presents first fundamental principles of the rheology of polymeric fluid including kinematics and stresses of a deformable body the continuum theory for the viscoelasticity of flexible homogeneous polymeric liquids the molecular theory for the viscoelasticity of flexible homogeneous polymeric liquids and the experimental methods for the measurement of the rheological properties of polymeric liquids The materials presented are intended to set a stage for the subsequent chapters by introducing the basic concepts and principles of rheology from both phenomenological and molecular perspectives of structurally simple flexible and homogeneous polymeric liquids Next this volume presents the rheological behavior of structurally complex polymeric materials including miscible polymer blends block copolymers liquid crystalline polymers thermoplastic polyurethanes immiscible polymer blends particulate filled polymers organoclay nanocomposites molten polymers with dissolved gas and thermosets

Modulated Temperature Differential Scanning Calorimetry Mike Reading, Douglas J. Hourston, 2006-02-22 MTDC provides a step change increase in the power of calorimetry to characterize virtually all polymer systems including curing systems blends and semicrystalline polymers It enables hidden transitions to be revealed miscibility to be accurately assessed and phases and interfaces in complex blends to be quantified It also enables crystallinity in complex systems to be measured and provides new insights into melting behaviour All of this is achieved by a simple modification of conventional DSC In 1992 a new calorimetric technique was introduced that superimposed a small modulation on top of the conventional linear temperature program typically used in differential scanning calorimetry This was combined with a method of data analysis that enabled the sample's response to the linear component of the temperature program to be separated from its response to the periodic component In this way for the first time a signal equivalent to that of conventional DSC was obtained simultaneously with a measure of the sample's heat capacity from the modulation The new information this provided sparked a revolution in scanning calorimetry by enabling new insights to be gained into almost all aspects of polymer characteristics This book provides both a basic and advanced treatment of the theory of the technique followed by a detailed exposition of its application to reacting systems blends and semicrystalline polymers by the leaders in all of these fields It is an essential text for anybody interested in calorimetry or polymer characterization especially if they have found that conventional DSC cannot

help them with their problems *Advanced Science and Technology of Sintering* Biljana D. Stojanovic,Valery V. Skorokhod,Maria Vesna Nikolic,2011-06-28 This volume entitled *Advanced Science and Technology of Sintering* contains the edited Proceedings of the Ninth World Round Table Conference on Sintering IX WRTCS held in Belgrade Yugoslavia September 1 4 1998 The gathering was one in a series of World Round Table Conferences on Sintering organised every four years by the Serbian Academy of Sciences and Arts SASA and the International Institute for the Science of Sintering IISS The World Round Table Conferences on Sintering have been traditionally held in Yugoslavia The first meeting was organised in Herceg Novi in 1969 and since then they have regularly gathered the scientific elite in the science of sintering It is not by chance that at these conferences G C Kuczynski G V Samsonov R Coble Ya E Geguzin and other great names in this branch of science presented their latest results making great qualitative leaps in the its development Belgrade hosted this conference for the first time It was chosen as a reminder that 30 years ago it was the place where the International Team for Sintering was formed further growing into the International Institute for the Science of Sintering The IX WRTCS lasted four days It included 156 participants from 17 countries who presented the results of their theoretical and experimental research in 130 papers in the form of plenary lectures oral presentations and poster sections **1991 International Conference on Coal Science Proceedings** Sam Stuart,2013-09-17 1991 International Conference on Coal Science Proceedings Thermal Physics and Thermal Analysis Jaroslav Šesták,Pavel Hubík,Jiří J. Mareš,2017-03-24 Features twenty five chapter contributions from an international array of distinguished academics based in Asia Eastern and Western Europe Russia and the USA This multi author contributed volume provides an up to date and authoritative overview of cutting edge themes involving the thermal analysis applied solid state physics micro and nano crystallinity of selected solids and their macro and microscopic thermal properties Distinctive chapters featured in the book include among others calorimetry time scales from days to microseconds glass transition phenomena kinetics of non isothermal processes thermal inertia and temperature gradients thermodynamics of nanomaterials self organization significance of temperature and entropy Advanced undergraduates postgraduates and researchers working in the field of thermal analysis thermophysical measurements and calorimetry will find this contributed volume invaluable This is the third volume of the triptych volumes on thermal behaviour of materials the previous two receiving thousand of downloads guaranteeing their worldwide impact *Climate Change, Carbon Capture, Storage and CO2 Mineralisation Technologies* Nikolaos Koukoulzas,Pavlos Tyrologou,Petros Koutsovitis,2020-12-04 This Special Issue delivered 16 scientific papers with the aim of exploring the application of carbon capture and storage technologies for mitigating the effects of climate change Special emphasis has been placed on mineral carbonation techniques that combine innovative applications to emerging problems and needs The aim of this Special Issue is to contribute to improved knowledge of the ongoing research regarding climate change and CCS technological applications focusing on carbon capture and storage practices Climate change is a global issue that is interrelated with the energy and

petroleum industry

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