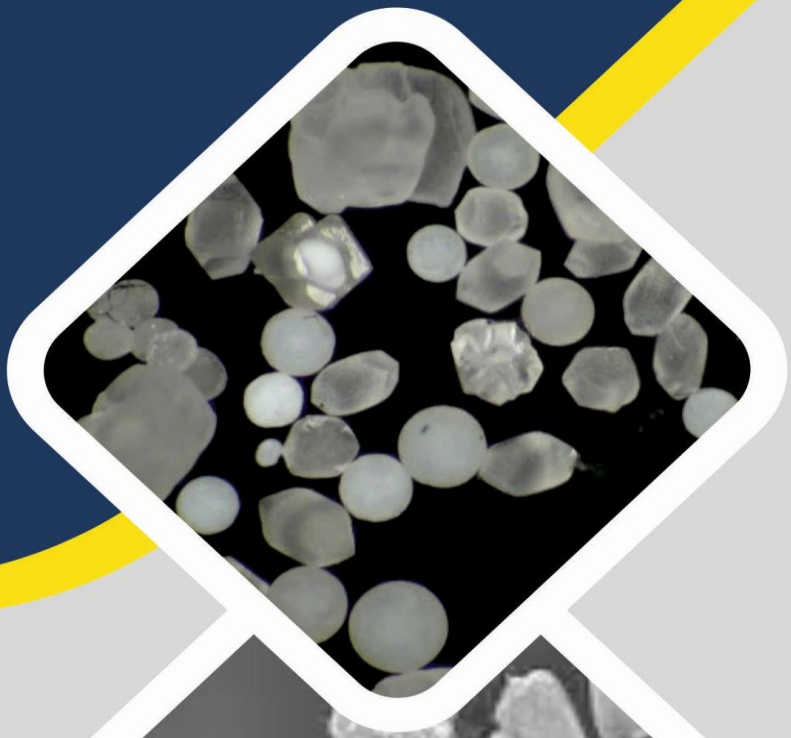
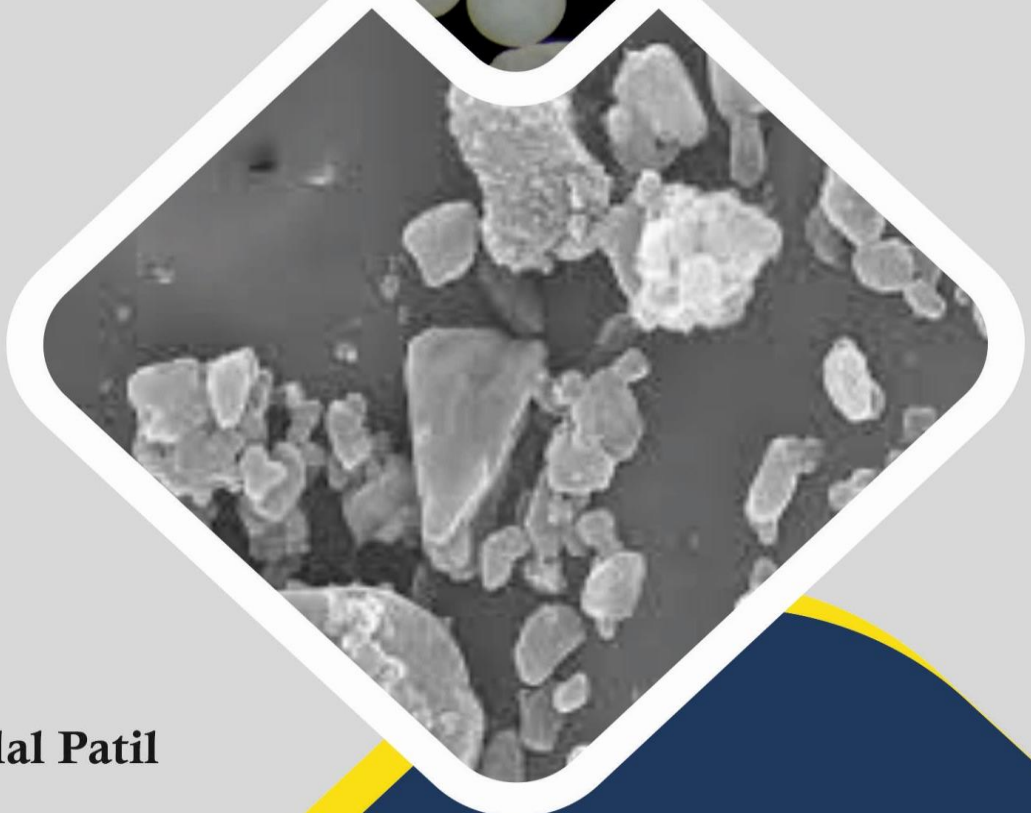
Characterization of Gel Grown Tartrate Crystals of Lanthanide Series Elements





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CHARACTERIZATION OF GEL GROWN TARTRATE CRYSTALS OF LANTHANIDE SERIES ELEMENTS

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PREFACE

Crystals have ever fascinated mankind. The growth of crystals occurs either in nature or artificially in a laboratory. The Mother Nature has grown a variety of crystals in the crust of earth, which are mainly diamond and other precious stones. Systematic study of the growth and properties of crystals is covered under the subject “Crystal Growth.” Today, the growth of crystals does not remain the phenomena only occurring in nature, but it has become a well advanced as well as widely used laboratory technique. There always has been a requirement of good quality crystals for various applications. In this regard the crystals having specific properties can be treated as the backbone of today's technological development. This led the investigators and scientists to concentrate on the developments of new variety of defect-free crystals of high degree of purity. Earlier crystal growth techniques were considered an art rather than science. Theories are now available on the growth of crystals so that crystal growth is no more an art. A variety of experimental techniques are developed and modified to such a level as to grow tailor made crystals for specific applications.

This book reports the detailed study on various physical properties of gel grown tartrate crystals of lanthanide series elements. Materials in the form of tartrate compounds deserves special attention because of their many interesting physical properties such as dielectric, piezoelectric, ferroelectric and optical second harmonic generation. The rare earth compounds have attracted considerable attention on account of their luminescent and magnetic properties. Among the rare earth compounds lanthanum, cerium and neodymium tartrate crystals were selected because of their promising technical applications in optics and magnetism. Beside this the lack of work on the growth of these materials prompted the initiation of this work.

This book is a comprehensive account of the characterization of lanthanum tartrate, cerium tartrate and neodymium tratrate crystals. It contains the observations and results of the characterization of lanthanum tartrate, cerium tartrate and neodymium tratrate crystals.

The scope of the book is straight forward and designed in five chapters.

Chapter 1 contains a brief note on the experimental techniques employed in the study. They include XRD, FTIR, SEM, EDAX, UV-spectroscopy, thermal analysis, surface characterization.

Chapter 2 deals with characterization of lanthanum tartrate crystals. All observations and findings are correlated to the theories. This chapter also presents the details of the systematic study of the characterization of the crystals. Characterization of the grown crystals has been done using different techniques. The minute surface details are examined using a recently developed technique, the scanning electron microscopy (SEM). X-ray analysis gives the nature of the crystallinity of the crystals and the cell parameters have been evaluated. The FT-IR spectroscopy and thermal studies (TGA, DTA, DTG, DSC) studies throw light to the functional group and molecular (chemical) formula of the crystals. The thermal analysis results obtained are discussed and it confirms the molecular formula. The percentage of incorporation of different rare earth ions were determined by energy dispersive X-ray analysis (EDAX). UV-vis spectroscopy results are discussed in detail. Pore size, Surface area, pore volume are obtained from surface characterization technique.

Chapter 3 gives a detailed account of the characterization of gel grown cerium tartrate crystals.

Chapter 4 covers the characterization of neodymium tartrate crystals in detail.

Chapter 5 devoted to the summary and comparative study of characterization of this gel grown crystals.

H.M. Patil

Dedicated To

My parents

For

Their Inspiration,

Their Warmth,

But Mostly

For

Their Ethics

Acknowledgement

I wish to express my deep sense of gratitudes to **Dr. Dilip. S. Bhavasar,** Department of electronics, Pratap College, Amalner, whose deep interest and inspiring guidance throughout the course of this investigation made this work possible.

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Finally, I would like to thank all the people who assisted directly or indirectlyfor the success of this work.

H. M. PATIL

Abstract

Pure crystals of lanthanum tartrate, cerium tartrate and neodymium tartrate were grown by the diffusion of aqueous solutions of lanthanum chloride, cerium chloride and neodymium chloride as an upper reactant into the set gel embedded with tartaric acid. The single diffusion gel growth technique was employed for the growth of these crystals. The growth conditions were optimized by varying various parameters such as gel density, pH, gel setting time, aging of the gel, concentration of the reactants and temperature. Crystals of various dimensions and morphologies were obtained. Most of them were platelet, acicular and spherulites in shape.

The as grown crystals were characterized by various characterization techniques. The surface morphology of the spherulites grown is illustrated by scanning electron microscopy. The X-ray powder diffraction studies were carried to find out lattice parameters, grain size and texture coefficient. The X-ray powder diffraction studies reveal that all these crystals are polycrystalline. FTIR spectra for these crystals show all the bands expected from metal tartrate with water of hydration. It is seen from the band assignment that each metal atom is coordinated with one –OH and one carboxylic group from a tartrate molecule.

The thermal behaviour of the grown material was studied using TGA, DTA, DTG and DSC. The thermal stability and decomposition pattern of the grown crystals was established. Thermogravimetric analysis supports the correctness of the suggested chemical formula of the grown crystals. The percentage weight loss calculations from the thermogram were supplemented by EDAX and FTIR. The decomposition pattern of these crystals is reported to be typical of a hydrated metal tartrate with the rare earth oxides as the stable residue.

The energy dispersive X-ray analyses (EDAX) establish the presence of heavy rare earth elements qualitatively and to a good extent quantitatively. In addition, the optical characteristics were studied by UV-visible spectral studies. Surface area, pore size and pore volume analysis of powder sample was carried out using automated gas sorption system. The findings of these techniques of characterization are in good agreement with those reported in the literature. The implications are discussed.

Abbreviations

LaT	Lanthanum tartrate
CeT	Cerium Tartrate
NdT	Neodymium Tartrate
TG	Thermogravimetric
TGA	Thermogravimetric analysis
DTA	Differential thermal analysis
DTG	Derivative Thermogravimetric / Differential scanning calorimetry
DSC	Differential scanning calorimetry
UV-vis	Ultraviolet-visible spectroscopy
XRD	X-Ray Diffraction
SEM	Scanning Electron Microscope
NLO	Nonlinear Optics
KDP	Potassium di-hydrogen phosphate
DKDP	potassium di-deuterium phosphate
TGS	tri-glycine sulphate
KAP	potassium acid phthalate
LAP	Lithium Arginine Phosphate
SMS	Sodium Metasilicate
RHT	Rubidium hydrogen tartrate
CHPD	Calcium Hydrogen Phosphate Dihydrate
SHP	Strontium Hydrogen Phosphate
(BHP)	Barium Hydrogen Phosphate (BHP)
FT-I R	Fourier Transform Infrared
CCD	Charge-Coupled Device
DH	Dollimore-Heal
BJH	Barrett, Joyner and Halenda
DFT	Density Functional Theory
t-plot	Statistical thickness method
DR	Dubinin-Radushkevic
BET	Brunauer, Emmett and Teller

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ABOUT THE BOOK

This book is intended for use as reference book for undergraduate, postgraduate and research scholars as well as the beginners who desire to work in the field of characterization of crystals grown by gel method. The book provides a complete and comprehensive material on various characterization techniques. The subject matter is divided into five parts in simple and lucid language. Easy to understand most of the difficult and intricate topics. Book provides detailed study on the characterization of gel grown Lanthanum Tartrate, Cerium Tartrate and Neodymium Tartrate crystals.

ABOUT THE AUTHOR

Dr. Hiralal Motilal Patil graduated from prestigious Pratap College, Amalner affiliated to Poona University, Pune in 1987 with First Class. He passed his M.Sc. degree examination in Physics from M.J. College, Jalgaon affiliated to Poona University in 1989 with First Class. He obtained M.Phil. Degree from Kavayitri Bahinabai Chaudhari North Maharashtra University, Jalgaon (Formerly known as North Maharashtra University, Jalgaon) in 1997 and now he holds a Ph.D. degree in Physics from a prestigious Kavaitri Bahinabai Chaudhari North Maharashtra University, Jalgaon and has conducted research at renowned institutions. He has 34 years of teaching experience at undergraduate level. Presently he is working as an Associate Professor at renowned Jijamata Education Society's Arts, Science and Commerce College, Nandurbar. He has authored one Text Book for undergraduate students. Dr. Hiralal Motilal Patil is a renowned researcher and scholar in the field of materials science and crystallography. He has made significant contributions to the development of novel crystal growth techniques, including the gel method. Their research focuses on understanding the properties and applications of gel-grown crystals, with a particular emphasis on thermal properties. Dr. Hiralal Motilal Patil has published numerous papers in esteemed scientific journals and has presented their work at various national and international conferences. He is actively involved in the scientific community; continue to advance the field of crystal growth by gel method through his innovative research.

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