T.C. (T.R.)
BOLU ABANT İZZET BAYSAL ÜNİVERSİTESİ
INSTITUTE OF GRADUATE STUDIES
DEPARTMENT OF CHEMISTRY

STUDY OF THE INFLUENCE OF THREE DIFFERENT COMPLEXING AGENT ON THE SOL-GEL FABRICATION AND OPTICAL PROPERTIES OF CE ION DOPED STRONTIUM ALUMINATE

MASTER OF SCIENCE, CHEMISTRY

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ACCEPTANCE AND APPROVAL PAGE

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ÖZGÜR ERİŞMİŞ

PREFACE

This thesis marks the conclusion of my extensive research and academic endeavors over the past years. It delves into the **Study of the Influence of Three Different Complexing Agent On The Sol-Gel Fabrication And Optical Properties of Ce Ion Doped Strontium Aluminate**, a subject that holds significant personal and professional importance to me. The insights and findings presented herein are the outcome of countless hours of rigorous experimentation, detailed analysis, and critical thought.

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ABSTRACT

STUDY OF THE INFLUENCE OF THREE DIFFERENT COMPLEXİNG AGENT ON THE SOL-GEL FABRICATION AND OPTICAL PROPERTIES OF CE ION DOPED STRONTIUM ALUMINATE
MSC THESIS
ÖZGÜR ERİŞMİŞ
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Inorganic luminescent materials in most cases consist of rare-earth ion embedded in the crystalline matrix. One of the widely studied matrices is the group of strontium aluminates. In $SrO–Al_2O_3$ system, there are many possible phases, namely $Sr_3Al_2O_6$, $SrAl_2O_4$, $SrAl_4O_7$, $SrAl_{12}O_{19}$, $Sr_4Al_2O_7$, $Sr_4Al_{14}O_{25}$, $Sr_{12}Al_{14}O_{33}$ and $Sr_{10}A_{l6}O_{19}$, as described in the literature. A lot of attention has been concentrated on $Sr_4Al_{14}O_{25}$ phase. In this study, $Sr_4Al_{14}O_{25}$ was synthesized via the sol-gel method using boric acid (H_3BO_3) as an auxiliary agent at different temperatures. Boric acid was found to lower the synthesis temperature, shorten calcination time, aid crystallization, and stabilize phases. Cerium (Ce), a rare earth metal, was then doped into the structure at varying concentrations and holding times. Three complexing agents—ethylene glycol (EG), ethylenediaminetetraacetic acid (EDTA), and citric acid (CA)—were used separately to evaluate their effects on the synthesis process. Structural analysis using XRD data showed that none of the agents altered the base structure of strontium aluminate. However, optical properties varied: citric acid resulted in the highest absorbance, while ethylene glycol led to the highest reflectance. These findings contribute to understanding the role of complexing agents in tuning optical behavior in Ce-doped strontium aluminates.

KEYWORDS: $Sr_4Al_{14}O_{25}$, Sol-gel, XRD, FTIR, UV-Vis

ÖZET

ÜÇ FARKLI KOMPLEKS OLUŞTURUCU MADDENİN CE İYON KATKILI STRONSİYUM ALÜMİNATIN SOL-JEL ÜRETİMİ VE OPTİK ÖZELLİKLERİ ÜZERİNDEKİ ETKİSİNİN İNCELENMESİ
YÜKSEK LİSANS TEZİ
ÖZGÜR ERİŞMİŞ
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İnorganik ışıldayan malzemeler, çoğunlukla, kristal matris içine gömülü nadir toprak iyonlarından oluşur. Yaygın olarak incelenen matrislerden biri stronsiyum alüminat grubudur. $SrO–Al_2O_3$ sisteminde literatürde açıklandığı gibi $Sr_3Al_2O_6$, $SrAl_2O_4$, $SrAl_4O_7$, $SrAl_{12}O_{19}$, $Sr_4Al_2O_7$, $Sr_4Al_{14}O_{25}$, $Sr_{12}Al_{14}O_{33}$ ve $Sr_{10}Al_6O_{19}$ olmak üzere birçok olası faz vardır. Bu fazlardan biri olan, $Sr_4Al_{14}O_{25}$, fazına yoğun ilgi vardır. Bu çalışmada, $Sr_4Al_{14}O_{25}$ bileşiği sol-jel yöntemiyle ve yardımcı madde olarak borik asit (H_3BO_3) kullanımı ile farklı sıcaklıklarda sentezlenmiştir. Borik asidin, sentez sıcaklığını düşürdüğü, kalsinasyon süresini kısalttığı, kristalleşmeyi desteklediği ve faz stabilizasyonu sağladığı belirlenmiştir. Daha sonra, yapıya farklı derişim ve bekletme sürelerinde nadir toprak metallerinden seryum (Ce) katkılanmıştır. Sentez sürecinde üç farklı kompleksleştirici ajan—etilen glikol (EG), etilendiamin tetraasetik asit (EDTA) ve sitrik asit (CA)—ayrı ayrı kullanılarak yapıya etkileri incelenmiştir. Yapısal analizler, bu ajanların stronsiyum alüminatın temel yapısını değiştirmediğini göstermiştir. Ancak optik özelliklerde farklılıklar gözlemlenmiştir: sitrik asit ile elde edilen numuneler en yüksek absorbans değerlerini verirken, etilen glikol kullanılan örnekler en yüksek yansıma (% reflectance) değerlerine ulaşmıştır. Bu sonuçlar, Ce katkılı stronsiyum alüminatların optik özelliklerinin kompleksleştirici ajanlara bağlı olarak değişebileceğini göstermektedir.

ANAHTAR KELİMELER: $Sr_4Al_{14}O_{25}$, Sol-gel, XRD, FTIR, UV-Vis

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LIST OF ABBREVIATIONS AND SYMBOLS

CA : Citric Acid

DTA : Differential Thermal Analysis

EDTA : Ethylenediaminetetraacetic Acid

FT-IR : Fourier Transform Infrared Spectroscopy

SEM : Scanning Electron Microscopy

TG : Thermogravimetry

UV-Vis : Ultra Violet-Visible

XRD : X-Ray Diffraction

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In addition, the author wishes to express his profound appreciation to his **family**, whose constant support, patience, and encouragement have been an invaluable source of strength throughout this academic journey. Their unwavering belief in the author's potential and their sacrifices, both seen and unseen, have made the successful completion of this thesis possible. Without their emotional support and unconditional love, this endeavor would not have reached its full realization.

1.INTRODUCTION

Inorganic luminescent materials are commonly composed of rare-earth ions embedded within a crystalline matrix. Among the widely studied matrices, strontium aluminates are particularly notable. The $SrO–Al_2O_3$ system features various potential phases, including $Sr_3Al_2O_6$, $SrAl_2O_4$, $SrAl_4O_7$, $SrAl_{12}O_{19}$, $Sr_4Al_2O_7$, $Sr_4Al_{14}O_{25}$, $Sr_{12}Al_{14}O_{33}$, and $Sr_{10}Al_6O_{19}$, as documented in the literature (Douy et al., 2003).

In recent years, considerable focus has shifted to the $Sr_4Al_{14}O_{25}$ phase, characterized by an orthorhombic Pmma space group structure with lattice parameters a = 24.7451 Å, b = 8.4735 Å, c = 4.8808 Å, and a unit cell volume of V = 1023.41 $Å^3$ (D. Wang et al., 1999). Capron et al. demonstrated that this phase can be synthesized at 1134 °C and remains stable up to 1500 °C (G. Blasse et al., 1994).

$Sr_4Al_{14}O_{25}$: Eu has gained attention for its application in white LEDs due to its greenish-blue emission at 495 nm when excited by nUV light and a quantum efficiency of 90% (G. Blasse et al., 1994). When co-doped with dysprosium, $Sr_4Al_{14}O_{25}$: Eu, Dy has been identified as a persistent phosphor with an afterglow exceeding 20 hours (S. Sharma et al., 2008; Y. Lin et al., 2001; Y. Lin et al., 2002; E. Nakazawa et al., 2006). Other dopants have also been explored within this phase. For instance, red phosphorescence has been observed in $Sr_4Al_{14}O_{25}$: Cr, Eu, Dy (R. Zhong et al., 2006) and $Sr_4Al_{14}O_{25}$: Mn (L. Chen et al., 2013). Compounds doped with samarium have shown orange-red emission (H. Luitel et al., 2016), while blue-green phosphorescence has been reported in $Sr_4Al_{14}O_{25}$: Eu, Tm, La (O. Manashirov et al., 2013). Although cerium-doped $Sr_4Al_{14}O_{25}$ phosphors have been studied (S. Han et al., 2006; S. Sharma et al., 2009), the inconsistent findings indicate the need for further research in this system.

1.1 The literature review on strontium aluminates

M. Peng et al. (2003) prepared $Sr_4Al_{14}O_{25}$:Eu^{2+} phosphors using the high-temperature solid-state synthesis method in an open atmosphere. They observed the reduction of Eu^{3+} to Eu^{2+} within the compound and explained the Eu^{3+} to Eu^{2+}

reduction process in an open atmosphere using a charge balance model. By conducting experiments on the model, they experimentally determined that the electrons located in the negative defect centers formed within the compound reduce Eu^{3+} ions to Eu^{2+} ions at a lower valence state.

Z. X. Yuan et al. (2004) synthesized $Sr_4Al_{14}O_{25}:Eu^{2+}$, Dy^{3+} phosphors using the solid-state synthesis method from different initial compositions. They found that the phase composition and emission spectrum were not affected by the variation in the initial composition. However, it was observed that the emission characteristics were influenced by changes in the initial composition. A longer-lasting emission was observed in the phase with an Al/Sr ratio of R=3.5, as the calculated trap depth and energy were deeper compared to the phase richer in strontium ions.

Y. Lin et al. (2002) synthesized $Sr_4Al_{14}O_{25}:Eu^{2+}$, Dy^{3+} phosphors and examined their luminescence properties. In the emission spectrum, two emission bands were observed at 407 nm and 494 nm, with the former being weaker and the latter more intense. The energy exchanges between Eu^{2+} as the emission center and the traps result in luminescence lasting for more than 20 hours.

C. Chang et al. (2004) synthesized and characterized nano-sized $SrA_{l2}O_4:Eu^{2+},Dy^{3+}$ and $Sr_4Al_{14}O_{25}:Eu^{2+},Dy^{3+}$ phosphors using the co-precipitation method and studied their luminescent properties. The phosphors exhibited emission peaks at 480 nm and 505 nm, respectively. Compared to phosphors synthesized using the solid-state method, a blue shift in the emission peaks was observed. This blue shift decreases as particle size increases. Both synthesized phosphors demonstrated prolonged luminescence.

A. Nag et al. (2004) investigated the impact of boric acid on the persistence of luminescence in Eu(II) and Dy(III) doped strontium aluminate phosphors doped. For this purpose, they synthesized $SrAl_2O_4:Eu^{2+},Dy^{3+}$ and $Sr_4Al_{14}O_{25}:Eu^{2+},Dy^{3+}$ phosphors and characterized them using IR, ^{27}Al-NMR, MASS spectroscopy, and X-ray powder diffraction (XRD) techniques.

S. H. Han et al. (2006) synthesized $Sr_4Al_{14}O_{25}:Eu^{2+}$ and $Sr_4Al_{14}O_{25}:Ce^{3+}$ phosphors using a solid-state reaction method, with H_3BO_3 as a flux. The $Sr_4Al_{14}O_{25}$ phase was successfully synthesized as a single phase by utilizing 80

mol% H_3BO_3. The optimal doping concentrations were determined to be 6 mol% Eu^{2+} and Ce^{3+}, respectively. In the photoluminescence spectrum, the Eu^{2+}-doped sample exhibited emission at 495 nm, while the Ce^{3+}-doped sample showed emission at 400 nm. Cathodoluminescence studies indicated that both phosphors could be excited at low voltages ranging from 200 to 300 V.

Georgobiani, et al. (2009) synthesized the phosphors of $Sr_4Al_{14}O_{25}:Eu^{2+},Dy^{3+}$ using the solid-state synthesis method and investigated their luminescence characteristics using 632.8 nm laser light. They analyzed the luminescence characteristics as a function of temperature and Cr^{3+} impurities. The inclusion of Cr^{3+} ions in the crystal lattice was identified by two sharp emission bands around the 700 nm region, which are characteristic of chromium ions. These emission bands originate from the 2E→4A2 transitions.

N. Suriyamurthy et al. (2008) examined the effect of cationic substitution on the photoluminescence and long-lasting luminescence characteristics of $Sr_4Al_{14}O_{25}:Eu^{2+},Dy^{3+}$ phosphors. They observed that phosphors with lower or higher strontium ion content exhibited increased photoluminescence intensity but found no correlation between long-lasting luminescence properties and non-stoichiometric composition. In non-stoichiometric phosphors, the peak emission wavelength shifted toward blue or green; however, the maximum of the long-lasting luminescence band remained unchanged by stoichiometry. At high calcium concentrations, the substitution of calcium for strontium resulted in white luminescence. Additionally, the photoluminescence emission centers and the afterglow emissions differed. Silver doping influenced luminescence intensity due to an increase in trap density.

In the past few years, there has been a significant rise in the availability of alkaline earth metals doped with rare earth metals because they have a stable structure, show fluorescence properties at high intensities, can be used for a long time, and have high quantum efficiency and stability (Springer Science, Business Media, LLC. Smets et al., 1989). Therefore, they also play a role in producing various devices, for example; optical memory units, televisions, security signs.(T. Matsuzawa et al., 1996; W. M. Yen et al., 2007). It is also known that these materials activated with cerium (Ce) rare earth metals have begun to be used in the

laser industry and high-density optical memory systems. (A.N. Georgobiani et al., 2009; G. Blasse et al., 1994).

$Sr_4Al_{14}O_{25}$ has an orthorhombic crystal structure and consists of regular octahedral AlO_6^- layers separated by a double layer of regular tetrahedral AlO_4^-. Additionally, there are two distict strontium sites in the $Sr_4Al_{14}O_{25}$ compound. The first is Sr1, which located in an oxygen multi-hedron region consisting of six oxygen atoms, while the second site is Sr2, which found in a complex multi-hedron region consisting of eight Oxygen atoms.

The phase structure of $Sr_4Al_{14}O_{25}$ has a three-dimensional network-shaped structure surrounding regular tetrahedral AlO_4^- and regular octahedral AlO_6^- layers consisting of channels in which Sr^{2+} ions are positioned in the a and c directions.

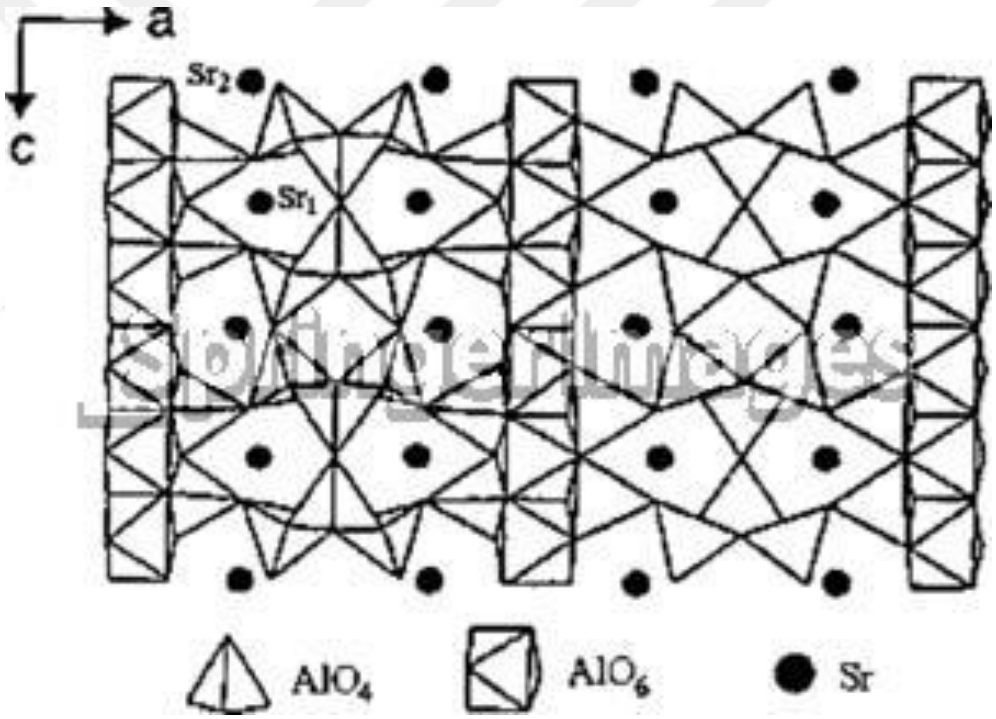

Figure 1.1. Crystal structure of $Sr_4Al_{14}O_{25}$

Aluminate guest matrices require high temperatures for inhomogeneous distributions and particle growth. For example, the fabrication of doped strontium aluminates was done via solid state method at 1400°C reported in (D. Haranath et

al., 2003) and (D. Wang et al., 1999). And, as researched, it was found that adding H_3BO_3 (boric acid) prior to the synthesis reduced the phase formation to 1300 °C.

Our aim in this study is to prepare cerium (Ce) rare earth metal doped strontium aluminate, $Sr_4Al_{14}O_{25}$ compound, using three different complexing agents, as it is in demand today for the above-mentioned purposes, and to examine the effects of these substances on determining the optical properties and present it to the literature to contribute to research on this subject.

1.2 Synthesis Methods

1.2.1 Solid-State Synthesis Method

Metal carbonates and/or oxides were mixed in appropriate stoichiometric ratios and ground using a ball mill. The resulting mixtures of starting materials were then fired in a horizontal tube furnace at high temperatures, either in open air or in a reducing gas atmosphere (%95 N2-%5 H2), to produce crystalline phosphor materials.

1.2.2 Hydrothermal Synthesis Method

In the hydrothermal synthesis method, metal salts or alkoxides are dissolved in an organic and/or polar solvent. The alkoxide compounds undergo hydrolysis in an aqueous and mildly acidic medium. Without undergoing gelation, the solution is transferred to a hydrothermal unit, where nano-sized powder products are obtained under varying temperatures and high pressures (Wang et al., 1999).

1.2.3 Pechini Sol-Gel Method

The Pechini sol-gel method is a well-known and straightforward technique for preparing metal oxide powders. In this process, metal salts are combined with a mixture of ethylene glycol and citric acid and heated at low temperatures to produce polymeric products. This method allows the formation of stoichiometric and molecular-level mixtures of metal cations by utilizing chelates formed between citric acid and metal cations in an aqueous solution. Moreover, this method is cost-effective and enables the production of materials with high-purity, homogeneous compositions, and ceramic films at low temperatures. The Pechini method is

commonly used for fabricating conductive materials, optical components, and more.

The Pechini process is preferred for the fabrication of specialized thin films such as conductive materials, optical materials, and ferroelectric materials (W.M. Yen et al., 1998).

1.2.4 Classical Sol-Gel Method

Sol-gel technology refers to a general technique used for the production of ceramics, glass, and composite materials for various applications, starting from a solution. Solutions are prepared by mixing metal alkoxides or inorganic compounds such as metal salts, oxides, hydroxides, and nitrates with specific amounts of acid and water. When this solution is heated to specific temperatures, it initiates a series of chemical reactions and electrochemical interactions among the surface charges of the particles in the solution. These interactions resulted in the formation of a network (gelation), that grows and eventually extends throughout the entire system, resulting in a complete structure (gel) (W.M. Yen et al., 1998).

The term "sol" describes a stable suspension of colloidal solid particles within a liquid. These particles of the solid must be sufficiently small to exhibit strong dispersion forces and maintain stability in the suspension.

A **colloid** is defined as particles that are too small to be seen with the naked eye, typically measuring 500 nm (1 nm = 10^{-9} m) or smaller. These particles cannot be observed with a standard optical microscope.

A **gel** refers to precipitates formed from colloidal particles, containing a large amount of water. Gel is considered an intermediate phase between solid and liquid states (Wang et al., 1999).

As noted by Hench LL (1990), sol-gel processing was first explored in the mid-1800s by Ebelman and Graham, who investigated silica gels in their efforts to synthesize inorganic ceramics and glass materials. In their experiments, tetraethyl orthosilicate ($Si(OC_2H_5)_4$) was hydrolyzed under acidic conditions, resulting in a glass-like substance composed of SiO_2. Fibers were extracted from the viscous gel, and composites were formed, but due to limited technological interest, a long drying process was required to prevent the SiO_2 gel from breaking into fine powder.

The initial successful application of the sol-gel method was carried out by Kistler SS (1932), who created SiO_2 aerogels using methanol and dried them through supercritical drying, forming a gel after solvent removal. Later, Roy R. (1956) developed an applicable phase diagram for ceramics using homogeneous powders. In 1969, Schroeder H. enhanced the optical characteristics of glass by incorporating SiO_2 and SiO_2-TiO_2 films into soda-lime glass coatings. The powder obtained through the sol-gel process was served as a precursor in sintering ceramics. many researchers made further advancements in the sol-gel method and the pioneering sol-gel workshop took place in the early 1980s, where the core concepts of sol-gel technology were outlined. Since then, the method has expanded to applications of organic-inorganic functional materials in diverse areas.

The sol-gel method primarily relies on two reactions: hydrolysis and condensation of precursors, which may be inorganic or metal-organic compounds such as alkoxides, acetates, nitrates, chlorides, formates, or acetylacetones dissolved in aqueous solutions. The sol-gel process involves multiple stages: hydrolysis, condensation, particle growth, gel formation, and drying, as illustrated in the schematic diagram below.

The starting materials are hydrolyzed to form a reactive monomer in the form of a colloidal solution, or sol. Typically, water, alcohol, and acid/base act as the hydrolysis agent, solvent, and catalyst, respectively. The process is followed by a polycondensation reaction in an aqueous solution, leading to the formation of a three-dimensional gelatinous network made up of either network polymers or discrete particles. During gelation, van der Waals forces, covalent bonds and hydrogen bonds form, with covalent bonds being dominant. This process is usually irreversible, although the reaction can be reversible if other interactions are involved (Muresan, LM, 2015; Rao, BG et al., 2017).

In addition, according to Aguilar G.V (2018), the particles in the gel have a polymeric substructure formed from the aggregation of sub-colloidal particles, while agglomerates of colloidal particles make up the gel network. The term "gel" was also discussed by Flory, who classified gels into several categories, including:

1. Systematic lamellar structures,
2. Disordered covalent polymeric networks,

3. Disordered polymer networks formed by physical aggregation
4. Distinct disordered structures.

Afterward, a thermal treatment is applied to remove the liquid phase, converting the gel phase into the final material after calcining at an appropriate temperature. The drying process can also affect the final phase of the product, resulting in either xerogels or aerogels. If a conventional drying method is used to remove the solvent, xerogels form, while supercritical drying leads to the formation of aerogels. Aerogels are known for their very low density and high thermal insulating properties, whereas xerogels have large surface areas. Consequently, the sol-gel method allows precise control over the reactions during synthesis, enabling control over the characteristics and structure of the resulting materials (Sakka, S. 2013; Aguilar GV, 2018; Sonawane, GH et al., 2018).

Control over the characteristics and structure of the gel can be easily achieved by adjusting hydrolysis and condensation conditions, changing process parameters such as temperature, pH, precursor and solvent nature, and adjusting the concentration of precursors. These parameters are essential for forming materials across a broad range of sizes, including nanomaterials, micromaterials, and macromaterials, and for optimizing properties like electrical, optical, and magnetic characteristics. Furthermore, the sol-gel method offers exceptional control over particle size distribution and structure (Itoh and Sugimoto, 2003; Aguilar GV, 2018).

The sol-gel technique is categorized into aqueous and nonaqueous methods, based on the solvent used during synthesis. In the aqueous sol-gel method, the oxygen needed for metal oxide formation is derived from water, with metal nitrates, acetates, chlorides, sulfates, and metal alkoxides as the starting materials. In contrast, in the nonaqueous sol-gel method, oxygen needed for metal oxide formation comes from ketones, aldehydes, alcohols, or metal precursors. While the aqueous sol-gel method is more commonly used and considered superior to the nonaqueous method, especially for nano-oxide formation, the nonaqueous method also offers its own set of advantages (Bradley et al., 2001; Turova et al., 2002; Song and Zhang, 2004).

In addition to its simplicity, the sol-gel method is sol-gel method is an efficient technique for synthesizing high-purity products at low temperatures while maintaining uniform nanostructures. The method also has several benefits: it is cost-effective, provides excellent crystallinity and homogeneity, offers precise composition control, allows for easy shaping of materials into complex geometries, and is suitable for large-scale synthesis (Muresan, LM, 2015; Prasad, S., 2018).

Sol-gel-derived products are used as advanced materials in a variety of forms, such as thin-film coatings, fibers, dense materials, ultrafine powders, porous materials, and aerogels. According to Sakka S. (2013), sol-gel-derived materials possess various functional properties, including electronic, chemical, thermal, biomedical, and mechanical functions. They are used in applications such as anti-reflective coatings, phosphors, protective coatings for automobile windows, lasers, photocatalysts, transparent conductors, and ferroelectric materials.

Advantages of the Sol-Gel Process Compared to Other Techniques

1. **Simplified Equipment Requirements:** Large and complex systems are unnecessary for carrying out reactions; small reaction vessels are sufficient.
2. **No Need for an Inert Atmosphere:** Reactions can occur without requiring an inert environment.
3. **Versatile Solvent Options:** The initial medium can be aqueous or organic. By controlling the parameters that influence the reaction, the properties of the final product can be tailored.
4. **Preparation of Homogeneous Multicomponent Systems:** The sol-gel process enables the formation of well-homogenized multicomponent systems, which is challenging to accomplish with other methods.
5. **Lower Operating Temperatures:** Due to the high reactivity of gels, the process can be conducted at significantly lower temperatures compared to conventional glass and ceramic methods.
6. **High-Purity Products:** The use of synthetic starting materials instead of natural minerals ensures the synthesis of products with high purity.
7. **Minimized Material Loss:** The low reaction temperatures help reduce material loss due to evaporation, resulting in more efficient use of resources.

8. **Versatile Material Synthesis:** It is possible to synthesize fibers, films, ceramics, coatings, and other materials tailored to specific applications.

1.3 Aim of The Work

This study focuses on the sol-gel synthesis of the $Sr_4Al_{14}O_{25}$ compound, doped with cerium (Ce), a rare earth metal, using three different complexing agents. Given the increasing interest in strontium aluminate-based materials for optical applications, the goal is to explore how the selected complexing agents influence the optical properties of the synthesized compounds. The outcomes of this research are intended to contribute valuable data to the literature and advance the understanding of luminescent materials.

2. MATERIALS AND METHODS

2.1 Materials

The materials used in the preparation of cerium (Ce) rare earth metal doped strontium aluminate, $Sr_4Al_{14}O_{25}$ compound are:

- Cerium nitrate hexahydrate, ($Ce(NO_3)_2.6H_2O$, 99.00%, Aldrich)
- Strontium nitrate hexahydrate ($Zn(NO_3).6H_2O$, 99.00, Merck)
- Aluminium nitrates nonahydrate $Al(NO_3)_3$.
- Citric acid monohydrate ($C_6H_8O_7.H_2O$, 99.5%, Merck)
- 1,2-ethanediol ($C_2H_6O_2$, 99.00%, Merck)
- EDTA ($C_{10}H_{16}N_2O_8$, 99.00%, Aldrich)
- Boric acid (H_3BO_3, 99.00%, Merck)
- Pottasium bromide (KBr, 99.99%, Merck) for the IR analysis

2.2 Method

In this thesis, the following method was used to synthesize the target substances. Strontium nitrate was first dissolved in acetic acid and then, Aluminate and Cerium substances were added and after waiting for 5 minutes with the mouth closed, the complexing agent was added. After all the liquid in the solution evaporated, the remaining solid part was kept in the oven for one day, and then the auxiliary substance (Boric acid) was added and mixed and placed in the oven at 900ºC for 8 hours. After this waiting period, the substance obtained was placed back in the oven at different temperatures to complete the synthesis.

2.3 Instrumentation

To examine the structures of the compounds obtained after the substance synthesis is completed, X-ray powder diffraction and infrared spectroscopy; UV-Visible diffusion reflectance methods were used to examine the optical properties.

2.3.1 XRD (X-Ray Diffractometer)

The X-ray diffraction analysis was carried out by Rigaku Multiflex X-ray diffractometer, which was configured to operate with CuKα radiation. The experimental setup employed a voltage range of 30-40 kV and 10-20 mA, with a wavelength of λ = 1.54056 Å. To obtain accurate diffraction patterns, the weight of the sample was carefully measured and optimized. Additionally, the scanning speed was set at 5° per minute, and the step size was adjusted to 0.016° to ensure high resolution during the analysis. This setup allowed for precise determination of the crystalline structure and phase identification of the synthesized material.

Picture 2.1. XRD (X-Ray Diffractometer)

2.3.2 FT-IR (Fourier-Transform Infrared Spectrophotometer)

The FTIR analysis was conducted to investigate the molecular vibrations and chemical bonding within the sample. The infrared spectra of the final products were obtained with Perkin Elmer Fourier Transform Infrared (FTIR) spectrometer. This advanced instrument, equipped with spectrum software, was utilized to capture detailed IR absorption data over a broad wavenumber range from 4000 to 400 cm^{-1}.

Picture 2.2. FTIR (Fourier-Transform Infrared Spectrometer)

2.3.3 UV-VIS (UV-Visible Spectrophotometer)

The optical characteristics of the synthesized materials were carefully examined by Perkin Elmer Lambda 35 UV-VIS Spectrometer. This spectrophotometer was used to measure absorbance and reflectance, as well as to record the corresponding spectra of the samples over a broad wavelength range from 200 nm to 900 nm.

Picture 2.3. UV-VIS (UltraViolet-Visible Spectrophotometer)

3.RESULTS

3.1. Determining the best conditions to obtain single-phase $Sr_4Al_{14}O_{25}$ crystal and proving it by characterizing it.

Figure 3.1 shows the decompositions and single phase formation in the synthesis of $Sr_4Al_{14}O_{25}$ with the auxiliary agent (H_3BO_3) at various temperatures. As can be seen from the figure, it would be correct to say that 1300 °C is the best temperature. Additionally, $Sr_3Al_2O_6$ and $SrAl_2O_4$ at 1000 °C. It was found that the $SrAl_4O_7$ and $SrAl_2O_4$ phases formed intensively at 1100 °C, while previous studies have shown that these phases gradually transformed into $Sr_4Al_{14}O_{25}$ after 1100 °C (Y. Lin et al., 2002). It has been observed that the substance, which maintains its stability up to 1500 °C, begins to decompose after this temperature and forms $SrAl_2O_4$ and $Sr_4Al_{12}O_{19}$ phases. Apart from these, $(SrAl_2O_4)_6SrO$ and $SrAl_2O_4$ phases were also encountered at 1400 °C, which may be due to a small impurity and is not suitable for a single phase crystal peak.

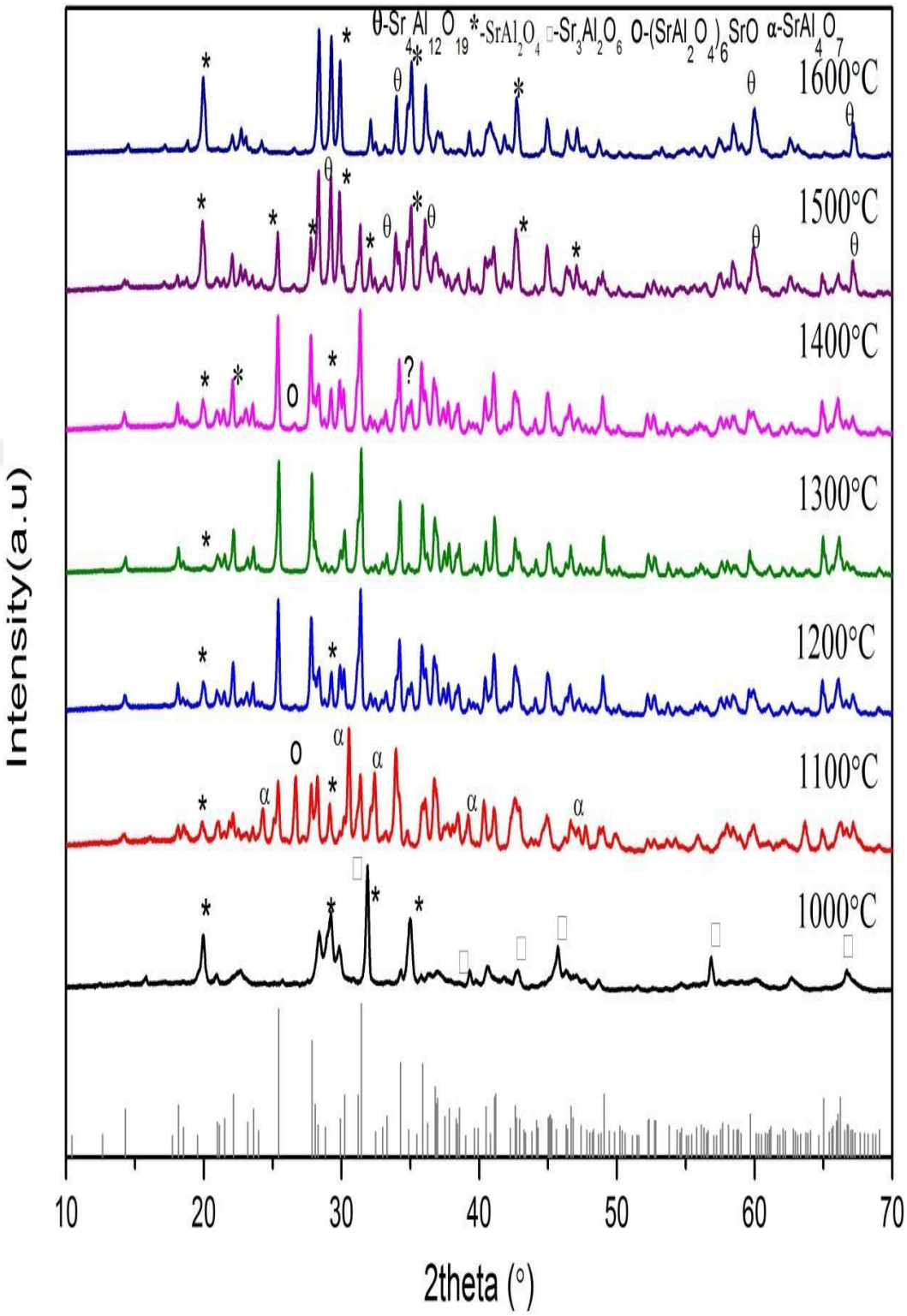

Figure 3.1. XRD values showing the single-phase crystal structure of $Sr_4Al_{14}O_{25}$ made with auxiliary agent (H_3BO_3) at different temperatures. Reference rods [00-074-1810] $Sr_4Al_{14}O_{25}$ ($Al_{10}O_{23}$).

Using these values and previous experiments, it can be said that the small amount of auxiliary added reduces the temperature, reduces the calcination time and helps crystallization. It has also been observed that it provides phase stabilization (E. Nakazawa et al., 2006).

3.2. Examination of the effects of Ce doping at different concentrations and keeping the material in the furnace for different time periods.

At this stage of the studies, we examined whether the substances synthesized under equal conditions (except for changes in Ce concentrations) at the same temperature (1300^{o}C) but different holding conditions (4 hours, 8 hours and 12 hours of heat treatment) would cause a change in the structure by analyzing the XRD data.

Accordingly, while there was no change in the structure of the material subjected to 4 hours and 12 hours of heat treatment, it was unexpectedly found that peaks such as Ce_2O_3 (except for the 0.005 concentration) in the structure of the material subjected to 8 hours of heat treatment disrupted the single-phase crystal structure. It was predicted that this was due to a personal mistake or impurity.

As a result of these data, it was determined that it was more appropriate to keep the substance in the oven for 4 hours (Figure 3.2) in terms of time saving, since it was observed that different waiting times did not cause a change in the structure.

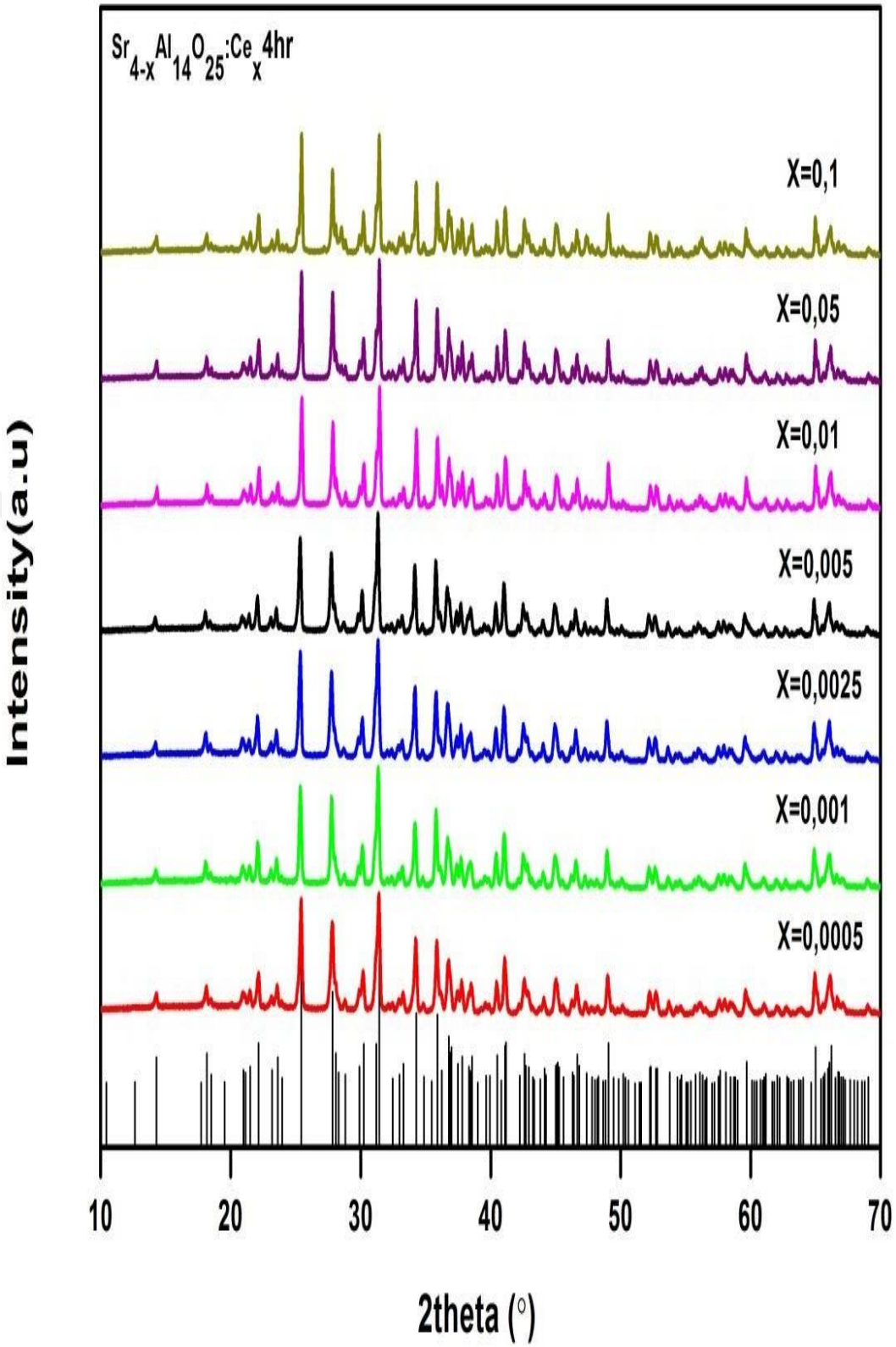

Figure 3.2. XRD results of Ce-doped $Sr_4Al_{14}O_{25}$ prepared by keeping it in the oven with auxiliary agent at 1300 °C for 4 hours, Reference rods, [00 -074-1810] $Sr_4Al_4O_2(Al_{10}O_{23})$

In terms of concentration; Increasing the concentration of Ce negatively affected the single-phase crystal structure and led to the emergence of peaks such as Ce_2O_3 (as at 0.2 M concentration). In addition, in the previous study of this substance using Luminescence, it was shown that Ce added with a concentration of 0.0025 M gave the most intense luminescence results, therefore 0.0025 M was preferred as the most appropriate concentration (R. Zhong et al., 2006; L. Chen et al., 2013).

3.3. Examining whether different complexing agents will bring about a change in the structure of the substance.

For this part, 3 different complexing elements (ethylene glycol, EDTA, and citric acid) were used. Whether there was a change in the structure of Ce-doped Strontium aluminate was examined using X-ray diffraction and infrared spectroscopy techniques and a comparison was made by visualizing it graphically.

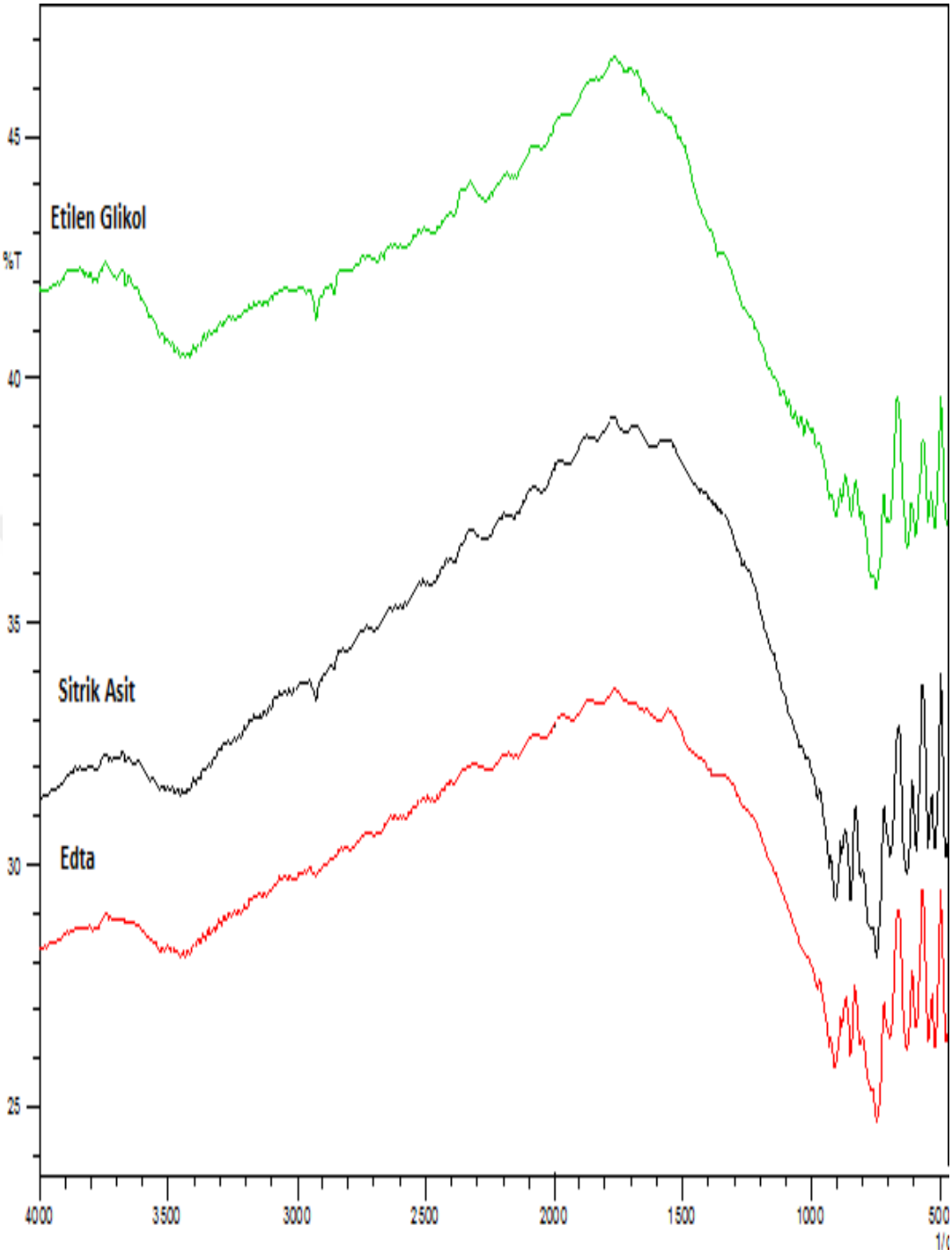

Figure 3.3. At 1300 °C, Ce-doped $Sr_4Al_{14}O_{25}$ becomes H_3BO_3 and three different Infrared spectra of the structures formed by the complexing agent

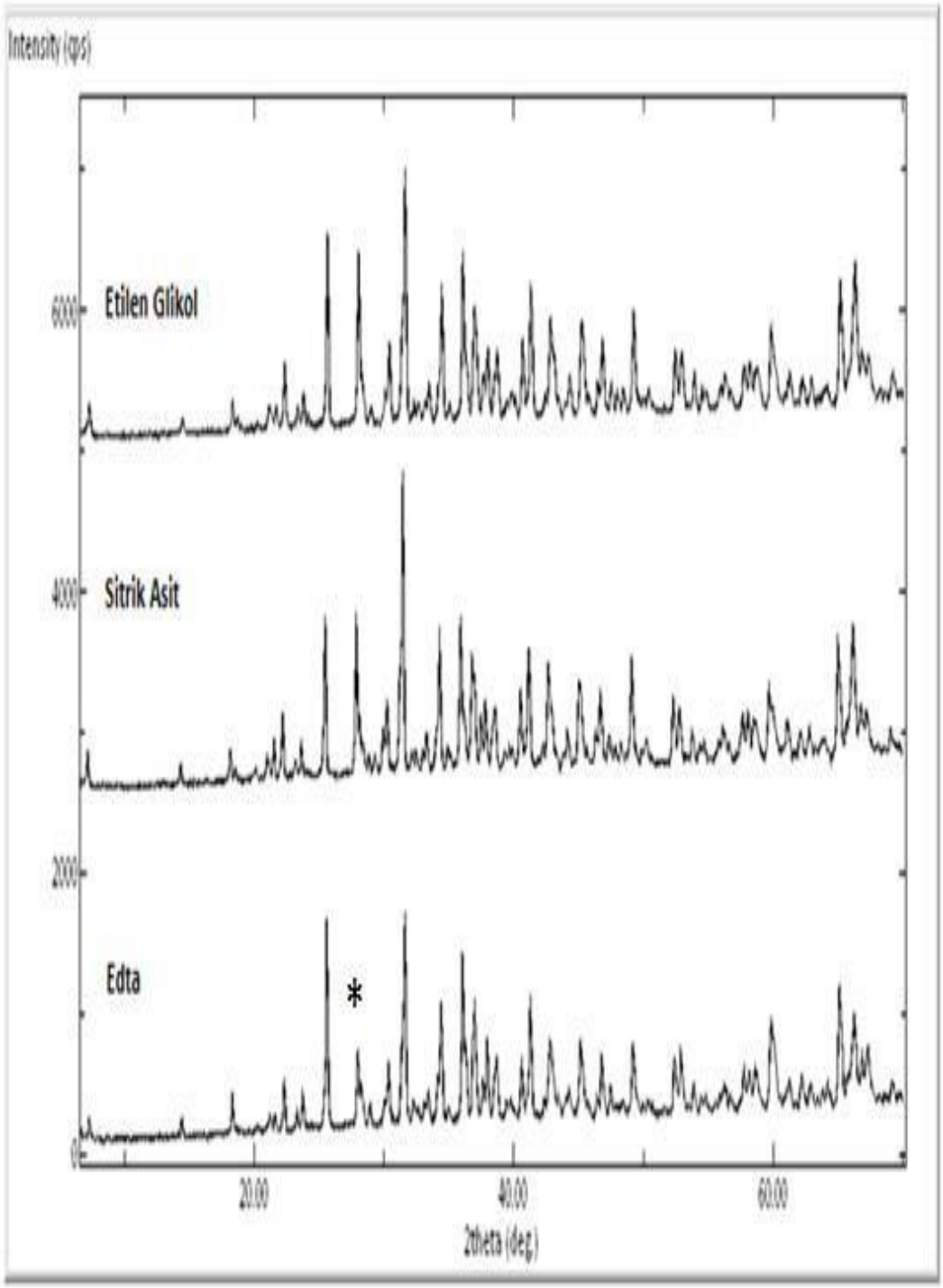

Figure 3.4. XRD data of the structures formed by Ce-doped $Sr_4Al_{14}O_{25}$ with auxiliary substance (H_3BO_3) and three different complexing agents at 1300 °C.

As a result of the analysis of the infrared spectra, no change was observed for the 3 different complexing substances, all infrared bands were completely similar to each other, and As a result of analyzing the XRD patterns, it was observed that the intensity of one peak in the synthesis involving the EDTA complexing agent was lower than that of the other peaks. However, it cannot be said that this detail causes a radical change in the structure of the matter. In short, it can be easily said, supported by quantitative results, that these three complexing groups chosen as the experimental group do not cause a change in the structure of strontium aluminate.

3.4. Study of the effect of different complexators on the optical properties of the material.

In the last part of the study, the optical properties of materials prepared with Ce-doped $Sr_4Al_{14}O_{25}$ at 1300 °C, along with an auxiliary material (H_3BO_3) and three different complexing agents which are citric acid, EDTA and ethylendiol were examined by the Diffuse Reflectance Spectroscopy, DRS, technique. For this purpose, the spectra of Ce-doped $Sr_4Al_{14}O_{25}$ prepared with three different complexing agents were recorded in the range of 200-900 nm using UV-Visible Spectrophotometer. The effect of three different complexing agents were observed during this analysis, and the optical band gap energy of the resulting products was determined.

The peaks were observed at wavelength below 400 nm in the spectra of cerium-doped $Sr_4Al_{14}O_{25}$ for all synthesized products with different complexing agents. The absorbance spectra were detected at the range of 200-400 nm. In comparison with the undoped strontium aluminate, the addition of cerium (III) ions as dopants to the host strontium aluminate, $Sr_4Al_{14}O_{25}$, compound has resulted in a clear and distinctive change in the optical properties of the host materials.

When Ce^{3+} is doped into strontium aluminate, $Sr_4Al_{14}O_{25}$, the observed absorption peaks in the ultraviolet (UV) region are primarily attributed to the 4f-5d electronic transitions of the Ce^{3+} ions. These transitions occur at high energy levels, corresponding to UV light absorption. While the strontium aluminate, $Sr_4Al_{14}O_{25}$, host lattice can affect the exact position and intensity of these peaks, the UV

absorption is fundamentally due to the intrinsic electronic properties of the Ce^{3+} ions, which dominate the spectral characteristics even within the host matrix.

As seen in Figures 3.5 and 3.6, it was observed that the cerium-doped strontium aluminate ceramic material obtained using citric acid gave the highest absorbance curves, but the material synthesized with ethylendiol gave the lowest absorbance curves.

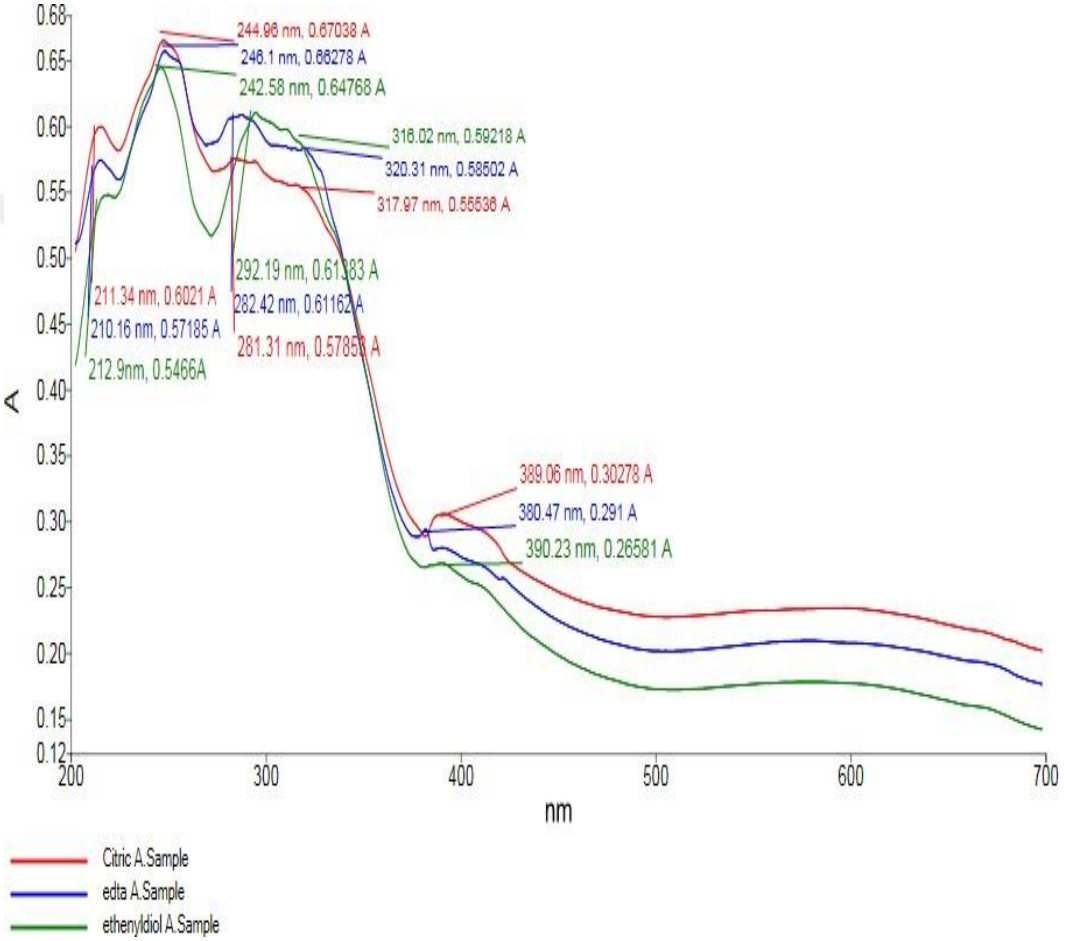

Figure 3.5. Absorbance data of Ce-doped $Sr_4Al_{14}O_{25}$ with auxiliary substance (H_3BO_3) and three different complexing agents at 1300 °C.

On the other hand, as a result of examining the % reflectance graphs, it was determined that the highest % reflectance values were seen in the materials synthesized with ethylendiol and the lowest % reflectance values were seen in the materials synthesized with the citric acid complexing element.

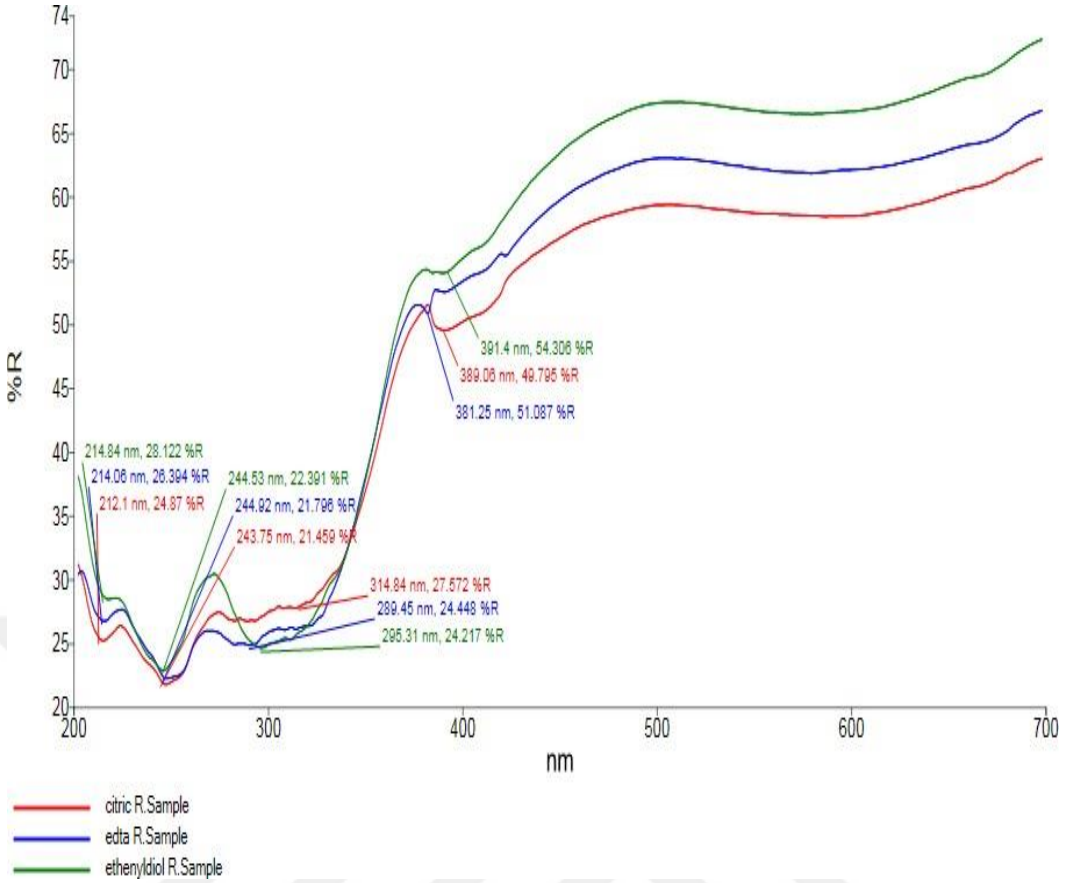

Figure 3.6. Diffuse reflectance curves of Ce-doped $Sr_4Al_{14}O_{25}$ with auxiliary agent (H_3BO_3) and three different complexing agents at 1300 °C.

The optical band gap energy of Ce-doped $Sr_4Al_{14}O_{25}$ at varying complexing agents was computed using UV-Visible absorbance data. This were achieved using a Tauc plot $((\alpha h\nu)^2$ versus energy) by taking the intercept of the curve on the energy in the x-axis (figure 3.7.) (S. Muthukumaran and R. Gopalakrishnan, 2012). Based on the measurements and calculations obtained, the band gap energy of Ce-doped $Sr_4Al_{14}O_{25}$ at varying complexing agents is in the range of 3.3255–3.3654 eV.

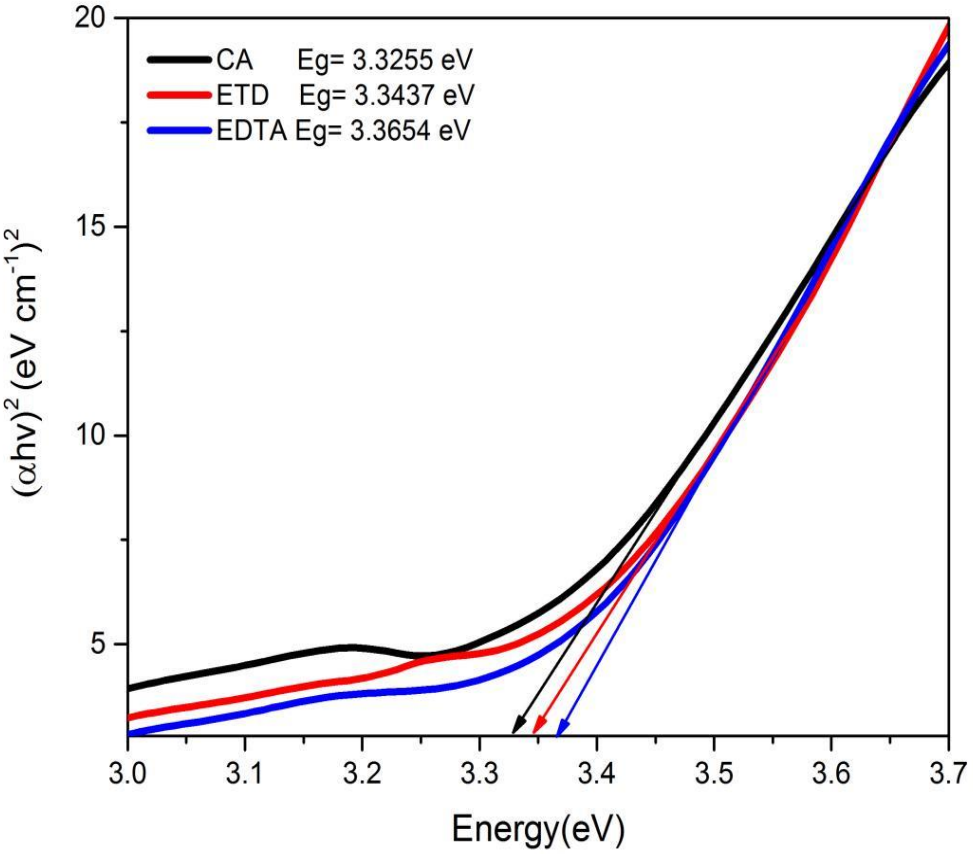

Figure 3.7. Band gap energies of Ce-doped $Sr_4Al_{14}O_{25}$ with auxiliary agent (H_3BO_3) and three different complexing agents at 1300 °C.

As a result of examining the band gap energies, it was determined that the highest band gap energy value was seen in the materials synthesized with EDTA and the lowest band gap energy value was seen in the materials synthesized with the citric acid complexing agent.

4. DISCUSSION

In the synthesis of $Sr_4Al_{14}O_{25}$ with the auxiliary agent H_3BO_3 at 1300 °C, various factors, such as Ce concentration and heat treatment time, were examined to assess their impact on phase formation and structural integrity. X-ray diffraction (XRD) analysis was used to investigate structural changes under different holding times (4, 8, and 12 hours) at the same temperature. The formation and stability of various phases in the Sr-Al-O system were examined at different temperatures. At 1000 °C, phases such as $Sr_3Al_2O_6$ and $SrAl_2O_4$ were observed. However, it was at 1100 °C that the $SrAl_4O_7$ and $SrAl_2O_4$ phases formed most intensively. Previous studies have shown that these phases gradually transition into $Sr_4Al_{14}O_{25}$ at temperatures above 1100 °C. Also, $Sr_4Al_{14}O_{25}$ remains stable up to 1500 °C, beyond which it begins to decompose. This decomposition results in the formation of $Sr_4Al_{12}O_{19}$ and $SrAl_2O_4$ phases. Additionally, at 1400 °C, the presence of phases including $(SrAl_2O_4)_6SrO$ and $SrAl_2O_4$ was also detected. These phases are likely due to small impurities, which is consistent with the observation that they do not contribute to a pure single-phase crystal structure

Based on the results, the material's structure remained stable with heat treatments lasting 4 and 12 hours, with no significant changes in phase composition. However, after 8 hours of heat treatment, the XRD patterns revealed the appearance of Ce_2O_3 peaks, indicating disruption of the single-phase crystal structure, except for the 0.005 M Ce concentration. This conditions was attributed to potential experimental errors or contamination. Based on these findings, a heat treatment duration of 4 hours was determined to be optimal, as it ensured phase stability without the risk of unwanted phase formation, thus saving time and resources.

Regarding Ce concentration, increasing the concentration negatively affected the single-phase crystal structure. At concentrations as high as 0.2 M, Ce_2O_3 peaks were observed, suggesting that excessive Ce incorporation disrupted the material’s phase. Furthermore, previous luminescence studies indicated that a Ce concentration of 0.0025 M resulted in the most intense luminescent output, making this concentration ideal for achieving both optimal luminescence and structural integrity.

These results emphasize the importance of carefully controlling both heat treatment time and Ce concentration when synthesizing $Sr_4Al_{14}O_{25}$ to obtain the desired structural and luminescent properties. Proper management of these factors is crucial for maintaining phase purity and ensuring the material's best performance.

Additionally, this study explored the influence of three different complexing agents such as ethylene glycol, EDTA, and citric acid on the structure and optical properties of Ce-doped $Sr_4Al_{14}O_{25}$. X-ray diffraction (XRD), infrared (IR) spectroscopy, and diffuse reflectance measurements were employed to assess these effects.

XRD analysis also confirmed that the overall crystal structure remained largely unaffected, although a slight decrease in the intensity of one peak was observed in the sample synthesized with EDTA. However, this minor change did not significantly disrupt the material's structure. The IR spectra showed no significant differences between the samples synthesized with different complexing agents, indicating that these agents did not cause any substantial changes in the chemical bonding or structure of $Sr_4Al_{14}O_{25}$.

Optical properties were evaluated using diffuse reflectance measurements. The Ce-doped material synthesized with citric acid exhibited the highest absorbance, indicating superior absorption in the UV-visible region. In contrast, the material synthesized with ethylene glycol showed the lowest absorbance. Furthermore, the % reflectance graphs revealed that materials synthesized with ethylene glycol had the highest reflectance values, while those synthesized with citric acid showed the lowest. These results suggest that citric acid enhances absorbance, while ethylene glycol increases reflectance, potentially making them suitable for different optical applications.

In conclusion, while the complexing agents did not significantly affect the structural integrity of $Sr_4Al_{14}O_{25}$, they played an important role in modulating its optical properties. Citric acid improved absorbance, while ethylene glycol enhanced reflectance. These findings demonstrate the potential for tailoring complexing agents to optimize the material’s performance for specific optical applications.

5. CONCLUSION

In this study, the Strontium aluminate, $Sr_4Al_{14}O_{25}$ compound was successfully synthesized via the sol-gel method, employing boric acid (H_3BO_3) as an auxiliary agent at varying temperature conditions. The primary aim of incorporating boric acid was to investigate its influence on the phase formation, crystallization behavior, and thermal processing conditions of the strontium aluminate matrix. The results revealed that boric acid significantly contributes to the formation of the $Sr_4Al_{14}O_{25}$ phase by acting as a fluxing agent, which promotes crystallization at comparatively lower temperatures. Furthermore, the use of H_3BO_3 was found to reduce both the required calcination temperature and the duration of heat treatment, thereby enhancing the efficiency of the synthesis process. Additionally, boric acid played a stabilizing role in preserving the desired phase, preventing the formation of undesirable secondary phases, and supporting the development of a well-defined crystalline structure.

Following the optimization of the base $Sr_4Al_{14}O_{25}$ matrix, cerium (Ce) ions were doped into the structure at various concentrations and holding durations to investigate the effect of Ce^{3+} incorporation on the material's structural and optical properties. Cerium, a rare-earth element known for its luminescent and charge transfer capabilities, was selected to enhance the optical functionality of the host material, with a particular interest in modifying its band gap and light absorption characteristics.

During the sol-gel synthesis of the Ce-doped $Sr_4Al_{14}O_{25}$ compounds, three different organic complexing agents—ethylene glycol (EG), ethylenediaminetetraacetic acid (EDTA), and citric acid (CA)—were employed to facilitate the chelation and uniform distribution of metal ions throughout the precursor solution. These chelating agents play a critical role in sol-gel processes, as they influence the homogeneity, gelation behavior, and ultimately the microstructure and purity of the final product. The study aimed to determine which of these agents contributed most effectively to the structural integrity and functional performance of the synthesized phosphor.

X-ray diffraction (XRD) analysis confirmed that the use of EG, EDTA, and CA as complexing agents did not lead to any notable changes in the crystal structure of the $Sr_4Al_{14}O_{25}$ compound. In all cases, the material maintained its single-phase structure, and no secondary crystalline phases were detected, indicating that the chelating agents do not interfere with the core crystallization process or lattice arrangement. This structural stability suggests that the $Sr_4Al_{14}O_{25}$ framework is tolerant to variations in the synthesis environment provided by different complexing agents.

However, despite the lack of structural variation, significant differences were observed in the optical properties of the Ce-doped materials depending on the chelating agent used. Ultraviolet-visible (UV-Vis) absorption spectroscopy showed that the sample synthesized using citric acid exhibited the highest absorbance intensity across the measured spectrum. This implies a greater light absorption capacity, which is advantageous for optical and photonic applications. In contrast, the sample prepared using ethylene glycol showed the lowest absorbance, indicating a lower interaction with incident light. Interestingly, the reflectance measurements showed an inverse relationship: the ethylene glycol-based sample exhibited the highest percentage reflectance, while the citric acid-based sample showed the lowest reflectance values. These findings suggest that citric acid not only enhances light absorption but may also influence surface morphology and particle dispersion in a manner that reduces reflectivity.

In addition to absorption and reflectance properties, the optical band gap energies of the synthesized phosphors were calculated from the Tauc plots derived from UV-Vis data. It was observed that the highest band gap energy was associated with the sample synthesized using EDTA, while the lowest band gap energy was found in the citric acid-assisted material. These differences are attributed to variations in electronic structure and localized states introduced by the choice of complexing agent, which can affect the degree of hybridization and orbital overlap between constituent atoms.

In summary, the study demonstrates that while the core crystal structure of $Sr_4Al_{14}O_{25}$ remains unaffected by the choice of complexing agent, the optical characteristics of Ce-doped samples are significantly influenced by the synthesis conditions, particularly the nature of the chelating agent. Citric acid, in particular, was shown to enhance light absorption and lower the band gap energy, making it a promising candidate for improving the optical performance of strontium aluminate-based materials. These findings provide valuable insights into the role of complexing agents in sol-gel chemistry and their impact on the functional properties of rare-earth doped luminescent materials. The outcomes of this study pave the way for further exploration of synthesis-structure-property relationships in phosphor materials, especially for applications in lighting, display, and optoelectronic technologies.

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