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Impact of annealing on structural and optical properties of sol-gel derived samarium silica nanocomposites

ABSTRACT

The pursuit of finely tuned material properties has driven the exploration of annealing strategies in the context of Samarium Silica Nanocomposites (Sm-SiO_2 NCs) synthesized through the sol-gel route. This study unveils novel insights into the influence of optimized annealing protocols on the structural and functional evolution of these advanced nanocomposites. Through meticulous experimentation, we establish that controlled temperature annealing plays a pivotal role in tailoring the microstructure and properties of Sm-SiO_2 NCs. The judicious manipulation of annealing parameters, including temperature duration, and atmosphere, orchestrates distinct transformations in the composite architecture. Field emission microscopy and structural analysis reveal that precise annealing promotes the consolidation of nanoscale domains, leading to improved crystallinity and enhanced connectivity between samarium species and the silica matrix. Moreover, the annealing-induced modifications extend beyond structural aspects to influence functional properties, an increase in crystallite size was observed from 15 nm to 43 nm as an effect of annealing. Our findings illustrate a remarkable enhancement in luminescence intensity as a consequence of optimized annealing, showcasing the potential for tailored photonic applications. These revelations are supported by a comprehensive suite of analytical techniques, including X-ray diffraction, Fourier transform infra red,\Field emission with Energy dispersive x-ray and photoluminescence spectroscopy. The synthesis-annealing synergy not only advances our fundamental understanding of nanocomposite evolution but also furnishes a pathway towards designing multifunctional materials with precision-engineered attributes.

Keywords: *Nanocomposites, Sol-Gel Route, annealing strategies, microstructural evolution, functional enhancements*

1. INTRODUCTION

In the pursuit of tailoring material properties at the nanoscale, the synthesis and optimization of multifunctional nanocomposites have gained substantial attention due to their potential applications in a wide array of fields. Among these, the coupling of rare earth elements with silica matrices has emerged as a compelling avenue for engineering advanced materials with tailored optical, magnetic, and catalytic properties [1-3]. The integration of Samarium Silica Nanocomposites (Sm-SiO_2 NCs) stands as a representative example, where the deliberate incorporation of samarium species into a silica matrix has shown remarkable potential for applications ranging from photonics to biomedicine. The sol-gel technique has emerged as a versatile and robust approach for fabricating nanocomposites with controlled compositions and morphologies [4,5]. In this context, the role of post-synthesis thermal treatments, particularly annealing, has garnered attention as a strategic tool for refining the properties of these nanocomposites. Annealing involves subjecting the synthesized materials to controlled elevated temperatures for a specified duration, often in controlled atmospheres [6,7]. Through this process, it is possible to induce structural rearrangements, phase transitions, and modifications in physicochemical properties that can be harnessed to enhance the functionality of the resulting nanocomposites [8-10].

This study delves into the realm of optimized annealing strategies for Sm-SiO_2 NCs prepared via the sol-gel route. The motivation behind investigating annealing stems from its potential to exert

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profound influence over the microstructure and properties of these nanocomposites [11,12]. By judiciously manipulating the annealing parameters, such as temperature, duration, and gas atmosphere, it becomes possible to drive controlled transformations that impact the arrangement of samarium species within the silica matrix. Consequently, these controlled changes in structure lead to corresponding enhancements in properties, such as photoluminescence intensity and magnetic behavior. The interplay between sol-gel synthesis and annealing presents an intriguing avenue for tailoring nanocomposite characteristics with precision [13-15]. Through a systematic exploration of annealing strategies, a deepened understanding of the structure-property relationships in Sm-SiO_2 NCs can be achieved. This investigation not only contributes to fundamental knowledge about nanocomposite evolution but also holds the potential to drive the development of innovative applications that capitalize on the synergistic effects of sol-gel synthesis and optimized annealing.

The annealing procedure plays a pivotal role in determining the final properties of the investigated Samarium Silica Nanocomposites prepared by the Sol-Gel method. In the subsequent sections of this paper, we present our findings on the influence of various annealing protocols on the structural and functional attributes of Sm-SiO_2 NCs. Through a combination of advanced characterization techniques, we unveil the intricate changes induced by annealing and underscore the potential of this approach for precise property engineering in nanocomposites.The study demonstrates that controlled temperature annealing is pivotal in tailoring the microstructure and properties of Sm-SiO_2 NCs. Optimized annealing protocols, including temperature, duration, and atmosphere, lead to distinct transformations in the composite architecture. This includes improved crystallinity, enhanced connectivity between samarium species and the silica matrix, and a remarkable enhancement in luminescence intensity. The findings highlight the significance of the annealing process in achieving precise property engineering in nanocomposites, advancing their potential applications in various fields.

2. MATERIALS AND METHODS

2.1. Materials

The materials employed for the preparation of Sm_2O_3-SiO_2 nanocomposites consisted of Tetraethyl orthosilicate, hydrochloric acid (HCl), nitric acid (HNO_3), samarium oxide and ethanol, all of which were acquired from Sigma Aldrich. Distilled water was utilized exclusively for all experiments conducted.

2.2. Methods

Using the sol-gel methodology, a silica gel doped with Sm was produced through refluxing highly pure chemicals. A mixture of tetraethoxysilane, ethanol, and deionized water was combined with hydrochloric acid, which acted as a catalyst. For the gel preparation, the molar ratios of starting solutions were adjusted as follows: 0.38:0.162:0.0923:0.031 for H_2O:C_2H_5OH:HCl:TEOS. Notably, all glass samples were doped with 6% Sm_2O_3. The inclusion of samarium oxide in the early stages of the process involved dissolving samarium oxide in nitric acid under elevated temperatures, yielding a clear white solution [16,17]. This resulting precipitate was then incorporated into the previously prepared solution. Following filtration, the solutions were stirred for a duration of 2h at room temperature, resulting in homogeneous solutions. Subsequently, these solutions were poured into various molds and placed in a drying oven set at 100°C. Gelation was evident approximately four days later. The aging process ensued, contributing to further contraction and solidification of the gel. The doped samples exhibited a glassy yellow hue due to the presence of samarium. To obtain powdered forms of the doped samples, a pestle and mortar were employed [18]. The resultant powder samples were subjected to sintering in a muffle furnace across temperature ranges of 780°C, 980°C, and 1180°C.

2.3. Characterizations

Characterizing the structural, morphological, and functional changes induced by optimized annealing strategies is pivotal to unravel the intricate interplay between thermal treatment and the properties of Samarium Silica Nanocomposites (Sm-SiO_2 NCs) synthesized via the sol-gel route. The characterization efforts provide a comprehensive understanding of how controlled annealing influences the evolution of these advanced nanocomposites. The characteristic diffraction peaks of samarium oxide and silica are meticulously tracked using RIGAKU XRD diffractometer. Energy-dispersive X-ray spectroscopy (EDS) coupled with FESEM allows for elemental mapping, facilitating the visualization of elemental distribution across the nanocomposite. Photoluminescence spectroscopy (PL) serves as a probing tool for assessing the impact of optimized annealing on the luminescent properties of Sm-SiO_2 NCs. FTIR has been used in order to trace out the functional groups attached herein with silica species.

3. RESULTS AND DISCUSSION

3.1. Crystallographic structure

The X-ray diffraction (XRD) analysis was conducted on samarium oxide-doped silica powder that underwent calcination at various temperatures (ranging from 780°C to 1180°C) for different durations as depicted in Fig.1. Interestingly, the XRD pattern of the powder subjected to calcination at 780°C for 3 h did not exhibit distinct reflection peaks, implying the material remained in an amorphous state. This outcome suggests that even after 3h of annealing well below the binary oxides' melting point, there was no discernible effect on altering the amorphous phase of the Sm_2O_3–SiO_2 composite. Upon increasing the calcination temperature to 980°C and maintaining it for 3 h, a pronounced transformation in the reflection pattern became evident. Notably, two prominent reflections emerged at angles 2θ of 28.22° and 32.75°. The broader peak centered around 2θ of 19.86° could be attributed to the (101) reflection of the cristobalite structure, as per the JCPDS file no. 39-1425. The presence of the cristobalite phase suggests the persistence of water molecules within the sample. Conversely, the sharp peak is plausibly associated with the (222) reflection of the cubic Sm_2O_3–SiO_2 phase. Notably, the application of heat treatment at 1180°C for 6 h led to a reduction in the number of pores and their interconnectivity, resulting in a substantial alteration of the amorphous phase [1, 9].

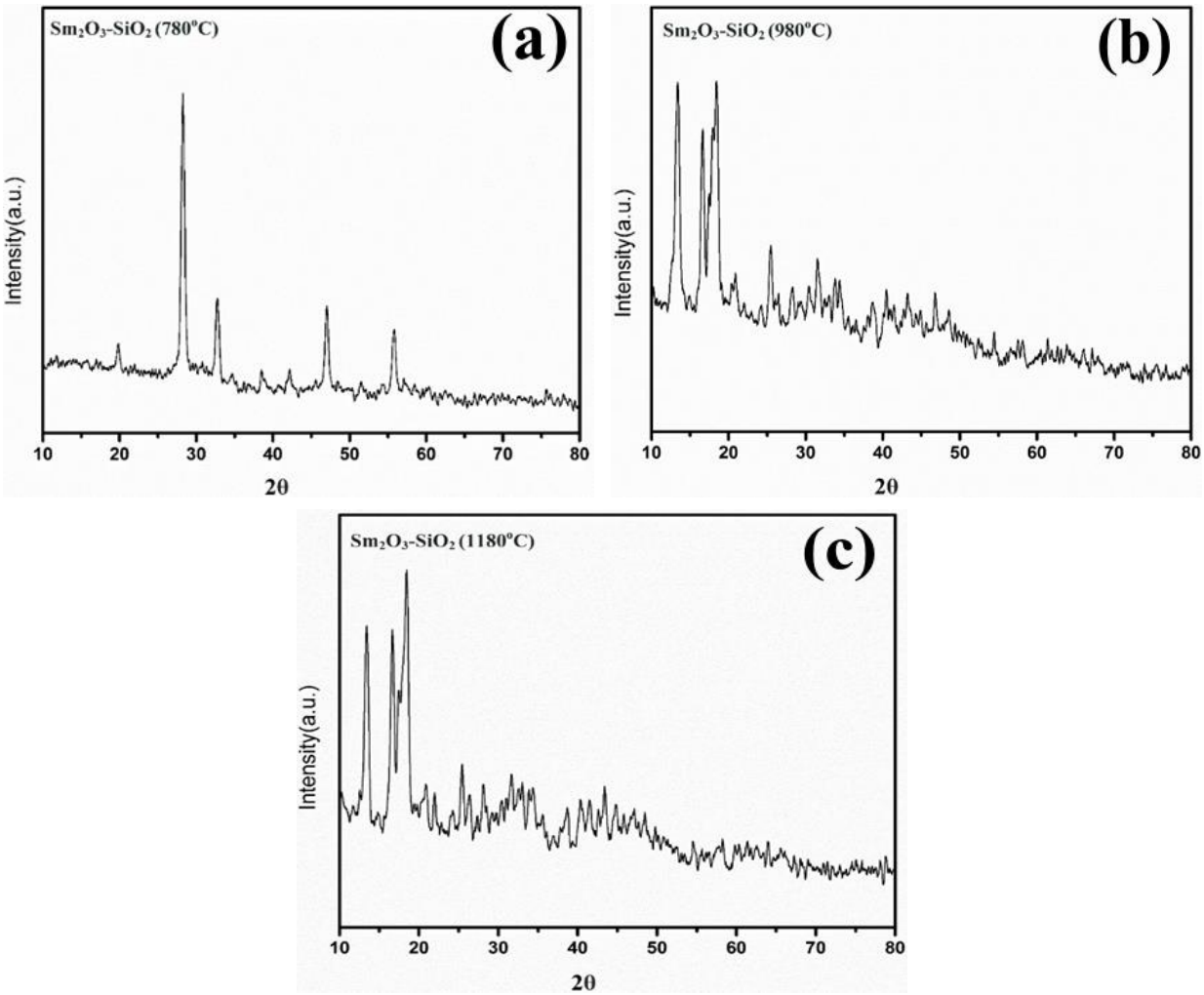

Figure 1. XRD pattern of annealed samples at (a) 780°C , (b) 980°C, (c) 1180°C

Slika 1. XRD grafik žarenih uzoraka na (a) 780°C, (b) 980°C, (c) 1180°C

It's noteworthy to mention that previous investigations did not reveal such significant reflections in the high Sm_2O_3 loaded sample annealed at 1000°C. However, annealing the sample at 780°C under vacuum conditions did yield a faint reflection. The narrower diffraction pattern around 28.22° was employed to calculate the mean crystallite size using the Scherrer formula, yielding

a size of 15 nm (with d=3.16 Å and a=10.94 Å). These outcomes suggest that during prolonged annealing for sintering, the crystallite size increases to 43 nm due to nanoparticle coalescence. These findings collectively indicate that low-temperature heat treatment (780-1180°C) combined with extended annealing duration enhances both the crystallinity and the size of the nanocomposites [6, 18]. These patterns not only ascertain the crystalline phases present but also reveal potential phase transitions triggered by annealing. The shift in peak positions, intensities, and widths unveils subtle changes in crystallographic parameters, shedding light on the arrangement of samarium species within the silica matrix.

3.2. FTIR

The FTIR transmittance spectra, spanning a range of 4000–500 cm^{-1}, vividly illustrate the effects of heat treatment on the doped samples. Notably, when subjected to a temperature of 980°C, distinctive bands emerge at 540, 800, 1629, and 1040 cm^{-1}. These bands find assignment in the symmetrical stretching vibration of Si–O–Si bonds, the vibrational mode of the ring structure of SiO_2 tetrahedra, the characteristic stretching mode Si–OH inherent to the gel structure, the TO mode originating from the asymmetric stretching vibration of Si–O–Si bonds, and the bending modes linked with water adsorbed onto the silica surface, respectively as shown in Fig. 2 [19, 20].

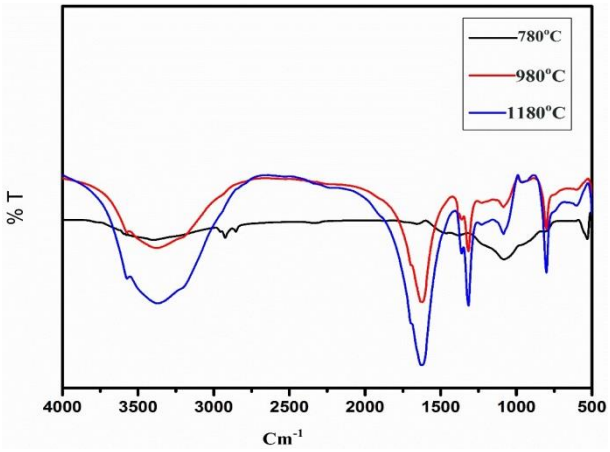

Figure 2. FTIR spectra of samarium silica nanocomposites

Slika 2. FTIR spektar nanokompozita samarijuma silicijum dioksida

In the lower frequency range of the FTIR spectra, a robust band centered around 1629 cm^{-1} is attributed to the Sm–OH bond. The intriguing facet lies in the transformation brought about by elevated temperature heat treatment, where the extended sintering process converts Sm–OH into the cubic Sm_2O_3 phase. Adding to this intrigue, the TO mode of Si–O–Si experiences a subtle shift towards higher wave numbers as the calcination temperature climbs to 1180°C during a 6 h treatment. The results obtained from FTIR analysis synergistically reinforce the findings deduced from X-ray diffraction (XRD) data [21]. The FTIR spectral shifts and distinctive bands align with the structural changes and transformations identified through XRD, collectively providing a robust validation of the dynamic alterations that transpire in the doped samples under varying heat treatment conditions [10].

3.3. Morphology of Sm_2O_3–SiO_2 nanoparticles

Field Emission Scanning Electron Microscopy (FESEM) coupled with Energy-Dispersive X-ray (EDX) analysis has unveiled profound insights into the microstructural and compositional changes exhibited by samarium-doped silica samples subjected to annealing at varying temperatures – 780°C, 980°C, and 1180°C as shown in Fig. 3 [15,21]. These observations provide a comprehensive picture of how heat treatment at different temperatures influences the morphology and elemental distribution within the composite material [2].

Annealing at 780°C: At this relatively lower annealing temperature, FESEM images reveal a predominantly agglomerated morphology of the doped silica particles. These clusters appear moderately porous, and the surface texture exhibits a level of irregularity. The particle sizes appear within the nanoscale range, with subtle variations attributed to agglomeration tendencies. The relative elemental composition reflects the intended composition of the composite as shown in Fig. 4.

Annealing at 980°C: Upon elevating the annealing temperature to 980°C, a noticeable transformation in the microstructure becomes evident. FESEM images portray a refined particle arrangement, with reduced agglomeration and a more uniform distribution. The surface texture appears smoother, with indications of enhanced particle connectivity.

Annealing at 1180°C: At the highest annealing temperature of 1180°C, FESEM images exhibit a further evolution in the microstructure. The particle agglomerations are significantly mitigated, leading to a more dispersed arrangement. The particle surfaces appear smoother and more homogenous, with indications of reduced porosity.

EDX analysis reaffirms the consistent presence of samarium, silicon, and oxygen. Interestingly, quantitative analysis of elemental proportions reflects a marginal change, potentially indicating subtle shifts in elemental diffusion due to the elevated annealing temperature.

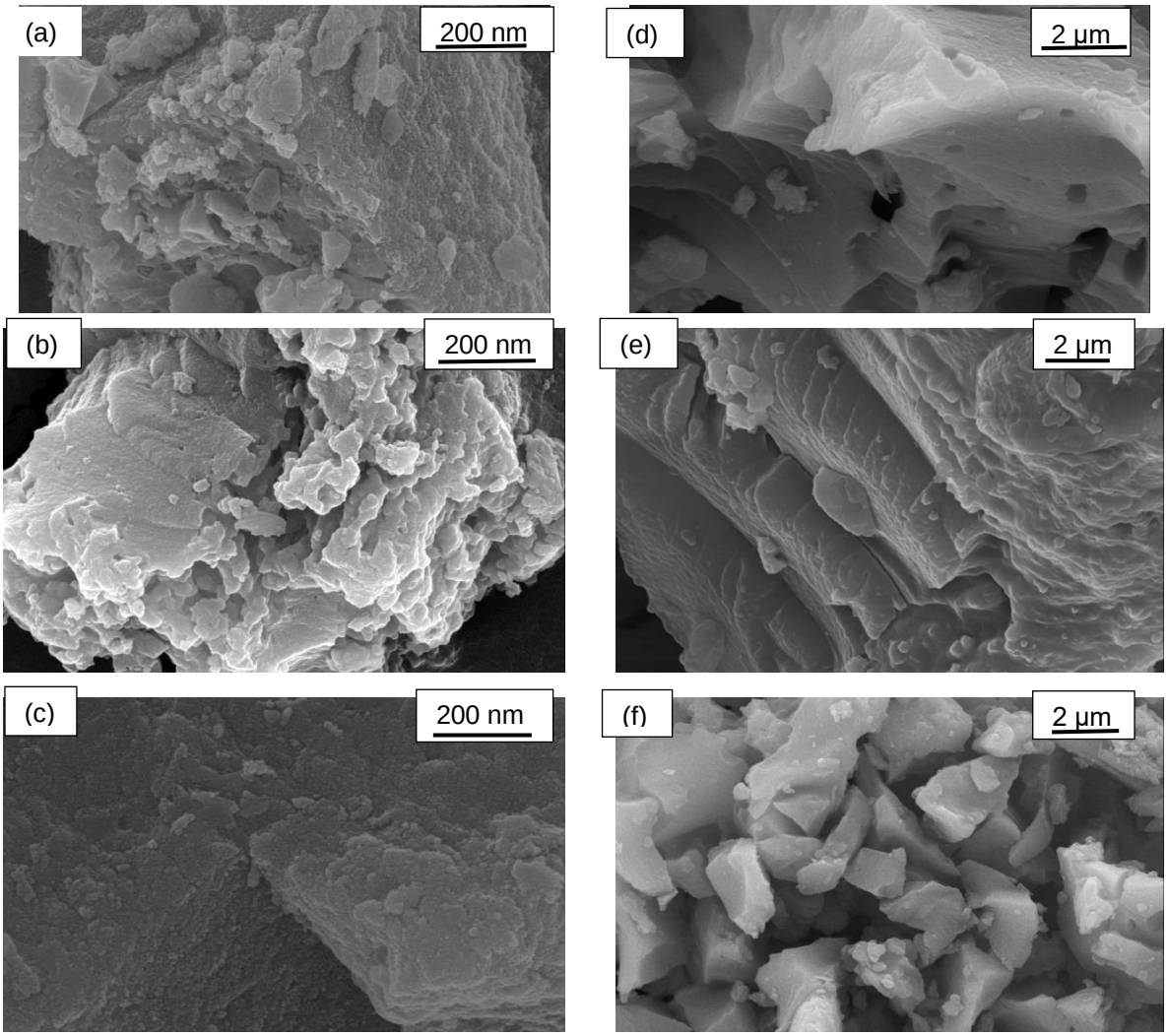

Figure 3. FESEM images of samarium silica nanocomposites annealed at 780°C, 980°C and 1180°C respectively; (a,b,c) at 200 nm scale and (d,e,f) at 2 µm scale

Slika 3. FESEM slike nanokompozita samarijum silicijum dioksida žarenih na 780 ^{0}C, 980^{0}C i 1180^{0}C respektivno; (a,b,c) na skali od 200 nm i (d,e,f) na skali od 2 µm

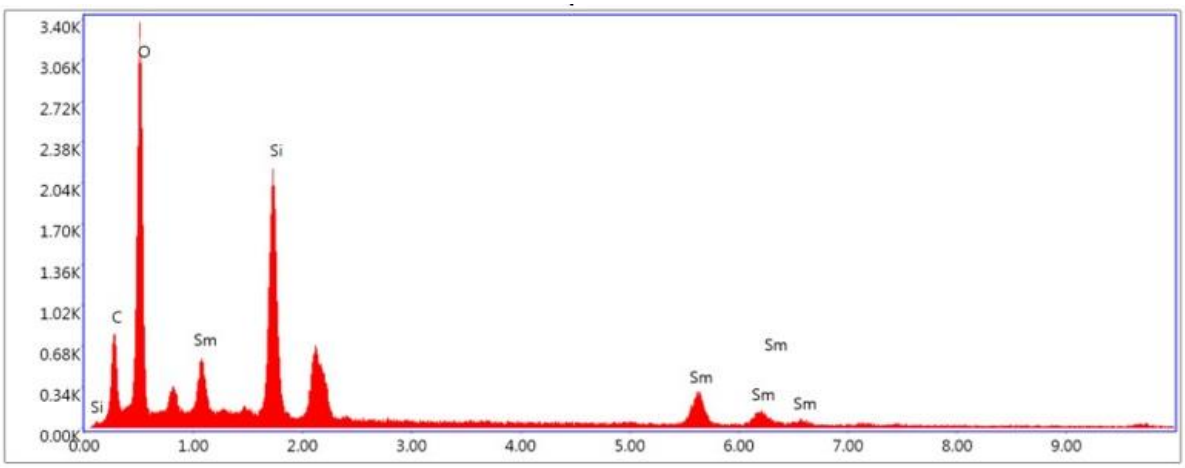

Figure 4. EDX spectra of samarium silica nanocomposites

Slika 4. EDX spektri nanokompozita samarijuma silicijum dioksida

Collectively, the FESEM images and EDX analyses illustrate a compelling narrative of structural refinement and compositional stability as the annealing temperature increases from 780°C to 1180°C [16]. The observed trends in morphology and elemental distribution bear significant implications for tailoring the material properties, influencing applications in catalysis, photonics, and beyond.

3.4. Photoluminiscence study

The presented Fig. 5 encapsulates the Photoluminescence (PL) spectra of typical powdered Sm_2O_3-SiO_2 samples, each undergoing distinct annealing conditions in ambient air. The samples are subjected to temperatures of 780°C (3 h), 980°C (3 h), and 1180°C (6 h). Notably, all spectra showcase continuous and broad luminescent patterns, characterized by a distinctive peak centered around 618 nm. This luminescence is attributed to the presence of samarium crystallites, despite the influence of concentration quenching [22, 23].

Of particular interest is the intriguing interplay between temperature and PL intensity. As the temperature is elevated, there is a discernible elevation in PL intensity. This phenomenon is attributed to the augmentation of nonradiative processes, which in turn diminish the efficacy of radiative recombination [24-26]. This relationship underscores the intricate balance between temperature-induced processes that either enhance or hinder the luminescence efficiency within the Sm_2O_3-SiO_2 samples.

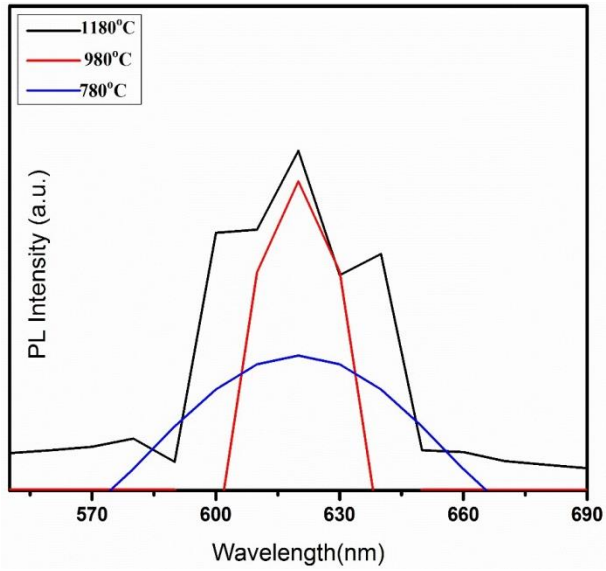

Figure 5. FTIR spectra of samarium silica nanocomposites

Slika 5. FTIR spektri nanokompozita samarijuma silicijum dioksida

4.CONCLUSIONS

The investigation has illuminated substantial alterations in both structural and functional attributes. Annealing-induced crystalline phase transitions and enhanced ordering underscore the pivotal role of temperature in shaping the material's architecture. Concurrently, the significant modifications in photoluminescence behavior emphasize the nuanced interplay between annealing and functional properties. One limitation may be the specific conditions chosen for annealing, as variations in temperature and duration could yield different results. Additionally, the research may focus on a particular aspect of the material properties, leaving room for exploration of other potential characteristics. Furthermore, the applicability of the findings in real-world scenarios or industrial settings may need further investigation. As for the future scope, expanding the research to encompass a broader range of annealing conditions and exploring diverse applications of Samarium Silica Nanocomposites could enhance the comprehensiveness of the study. Additionally, investigating potential synergies with other materials or incorporating advanced characterization techniques may open new avenues for understanding and optimizing the properties of these nanocomposites.

This study underscores the potential of annealing as a versatile tool for engineering tailored material characteristics. The synergy between sol-gel synthesis and annealing offers a promising avenue for creating advanced nanomaterials with finely tuned properties. As nanoscience advances, the insights garnered here contribute to the evolving landscape of material design, fostering opportunities for innovative applications across diverse technological domains.

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IZVOD

UTICAJ ŽARENJA NA STRUKTURNA I OPTIČKA SVOJSTVA NANOKOMPOZITA OD SAMARIJUM SILICIJUM DIOKSIDA DOBIJENIH SOL-GELOM

Potraga za fino podešenim svojstvima materijala dovela je do istraživanja strategija žarenja u kontekstu nanokompozita samarijum silicijum dioksida (Sm-SiO_2 NCs) sintetizovanih putem sol-gel puta. Ova studija otkriva nove vidike u uticaj optimizovanih protokola žarenja na strukturnu i funkcionalnu evoluciju ovih naprednih nanokompozita. Kroz pedantno eksperimentisanje, ustanovljeno je da žarenje na kontrolisanoj temperaturi igra ključnu ulogu u prilagođavanju mikrostrukture i svojstava Sm-SiO_2 NCs. Razumna manipulacija parametrima žarenja, uključujući trajanje temperature i atmosferu, orkestrira različite transformacije u kompozitnoj arhitekturi. Emisiona mikroskopija i strukturna analiza otkrivaju da precizno žarenje promoviše konsolidaciju domena nanorazmera, što dovodi do poboljšane kristalnosti i poboljšane povezanosti između vrsta samarijuma i matriksa silicijum dioksida. Štaviše, modifikacije izazvane žarenjem se protežu izvan strukturnih aspekata kako bi uticale na funkcionalna svojstva, uočeno je povećanje veličine kristalita sa 15nm na 43nm kao efekat žarenja. Naši nalazi ilustruju značajno poboljšanje intenziteta luminiscencije kao posledicu optimizovanog žarenja, pokazujući potencijal za prilagođene fotonske aplikacije. Ova otkrića su podržana sveobuhvatnim paketom analitičkih tehnika, uključujući difrakciju rendgenskih zraka, infracrvenu Furijeovu transformaciju, emisiju polja sa energetski disperzivnom rendgenskom spektroskopijom i fotoluminiscentnom spektroskopijom. Sinergija sinteze i žarenja ne samo da unapređuje naše fundamentalno razumevanje evolucije nanokompozita, već takođe pruža put ka dizajniranju multifunkcionalnih materijala sa precizno projektovanim atributima.

Ključne reči: *nanokompoziti, sol-gel ruta, strategije žarenja, mikrostrukturna evolucija, funkcionalna poboljšanja*

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