CHARACTERIZATION OF NiO NANOPARTICLES PREPARED USING GELATIN AND A LOW-COST SYNTHESIS

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Summary:

Nanocomposite material containing NiO nanoparticles decorated on WO_3 nanowires could be used for monitoring levels of H_2S gas. The effectiveness of this material is mostly determined by NiO properties. The performed study presents the first stage in the preparation of modified NiO/WO_3 nanocomposite material. In this article, NiO nanoparticles are prepared by a simple, low-cost method.The behavior of nano-sized NiO particles is probed by X-ray diffraction, Fourier transform Infrared Spectroscopy, Raman spectroscopy, and hysteretic measurement.

Key words: NiO, diffraction measurements, spectroscopy, nanoparticles, materials.

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Introduction

Transition metal (Fe, Co, Ni and Mn) oxide nanoparticles have been thoroughly examined in recent years due to discoveries of new applications of these materials (Sellmyer et al, 2001), (Jana et al, 2004), (Nikolić et al, 2016), (Nikolić et al, 2017), which is enabled by their modified properties that are significantly improved in comparison to bulk materials. Today, NiO nanoparticles are intensively investigated because of their various potential applications in different industries.

Nickel oxide materials are recognized as interesting for usage in catalytic reactions in industrial processes and electrochromic devices (Salimi et al, 2017), (You et al, 2003), (Xing et al, 2004), (Kamal et al, 2005), (Lee et al, 2014). One of potential applications of NiO nanoparticles in this field presents the fabrication of a glucose biosensor based on the electrochemical co-deposition of NiO and GOx (glucose oxidase) onto the surface of the GC (glucose consumption) electrode (Salimi et al, 2017). Further, the investigation of the influence of the NiO properties on the electrochemical properties of NiO/reduced graphene oxide-based supercapacitor electrodes is of importance for designing high performance energy storage devices constructed from materials with limited conductivity (Lee et al, 2014).

It is noteworthy that the examination of the properties of composite materials containing NiO nanoparticles could contribute to the development of inexpensive resistive sensors able to monitor the presence of H_2S in the environment, which is of significance for military industry. Jeffrey pointed to many potential hazards for workers involved in operation and maintenance of water and wastewater treatment systems (Jeffrey, 2015). Workers in navy air forces are often faced with the problem of the presence of explosive gases (such as methane (CH_4), carbon monoxide (CO) and hydrogen sulfide (H_2S)). In combination with the air and a spar, the mentioned gases are prone to strong explosions that could lead to significant damage in confined spaces (tanks, underground lift stations, manholes) (Jeffrey, 2015). Also, it is important to notice that in wastewater treatment systems, H_2S gas is present more often than any other hazardous gas (Jeffrey, 2015). After longer exposure, workers experience over-stimulation of all sensors and are not capable of registering H_2S odor. With the aim to overcome the mentioned problem, scientific community have performed more detailed research in H_2S sensors properties; literature data show that the behavior of H_2S sensors varies depending on the properties of material used for their construction (Rout et al, 2008), (Lin et al, 1994), (Wang et al, 2008), (Kapse et al, 2008), (MalekAlaie et al, 2015). Navarrete et al. found that tungsten oxide (WO_3) nanowires decorated with NiO nanoparticles could be applied onto substrates for developing resistive metal oxide gas sensors (Navarrete et al, 2018). These nanocomposite materials showed increased sensitivity and selectivity to H_2S, and achieved a five-fold increase in the response, which is ascribed to the NiO high chemical adsorption effect as well as to the high electronic sensitization effect (NiO nanoparticles become metallic Ni_xS_x upon exposure to H_2S).

The well-known gas sensing properties of the material consisting of nickel oxide (NiO) nanoparticles decorating tungsten oxide (WO_3) nanowires are significantly dependent on the properties of both present phases: NiO and WO_3 nanostructures. Although Navarrete et al obtained the mentioned material by performing a two-step synthesis (the first step presented the formation of pure tungsten oxide nanowires, and the second step considered loading these wires with 2 nm-sized NiO nanoparticles) (Navarrete et al, 2018), experience with other nanomaterials has revealed that the same material could be characterized by different properties if it is obtained by the modified synthesis procedure (Milić et al, 2017). Navarrete started the synthesis from expensive nickel(II)acetylacetonate (Navarrete et al, 2018) that is not appropriate for industry usage since industry always looks for low-cost solutions. One of potential synthesis approaches for the production of NiO/WO_3 nanocomposite material is to change the synthesis route performed for the preparation of NiO nanoparticles in order to estimate the best candidate for the discussed purpose.

Also, the literature review has revealed that it is not clear how variations in the size of NiO nanoparticles influence their chemical and electronic sensitization properties to H_2S. In Ref. (Navarrete et al, 2018), it is shown that the increased sensitivity and selectivity to H_2S is achieved by using 2 nm NiO nanoparticles. Having in mind that nanoparticles lower in size than 10 nm show different behavior compared to the nanoparticles of the same chemical composition but bigger in size (which is a consequence of more pronounced surface effects (Alkilany et al, 2009), (Cheng et al, 2006), (Kossyrev et al, 2005), (Müller et al, 2005), (Tramsdorf et al, 2007), it is difficult to predict whether NiO nanoparticles bigger than 10 nm will experience higher or lower sensitivity to H_2S gas detection, and this still has not been investigated in literature.

In order to investigate suitability of bigger NiO nanoparticles (~ 40 nm) for the detection of H_2S, this article presents the synthesis of NiO nanoparticles by a simple and low-cost coprecipitation method. This investigation is the first stage in the preparation of NiO/WO_3

nanocomposite material whose sensitivity and selectivity for H_2S will be investigated further in the future.

The characterization of the prepared NiO phase was performed by several techniques. X-ray diffraction (XRD) measurements were done by a Rigaku RINT-TTRIII using CuKα (λ = 1.5418 A). Diffraction patterns were recorded in the 2θ range (10-70°) with a scanning rate of 0.02 °/min. The Qualx Program was used for data analysis. Pure NiO nanoparticles were further investigated by Fourier transform infrared spectroscopy (Nicolet IS 50 FTIR Spectrometer, using the attenuated total reflectance (ATR) sampling technique). Raman spectroscopy was performed by micro Raman and the results analyzed using a Jobin–Yvon T64000 spectrometer equipped with a nitrogen cooled charge-coupled device detector. The measurement was done at 20 mW, using 532 nm green line as the excitation source, and the spectrum was recorded in back scattering geometry. Hysteretic measurement of the sample was carried out using a Quantum Design superconducting quantum interference device (SQUID). Hysteresis was recorded in a temperature range of 2 - 300 K for the applied magnetic fields from -50 to +50 kOe.

Experimental results and discussion

Synthesis of the samples

Two samples were prepared by a combination of the coprecipitation method and the annealing treatment (Maia et al, 2006). 6.365 g of nickel (II) chloride ($NiCl_2 \cdot 6H_2O$) and 15.913 g of commercial colorless gelatin were dissolved in 636 ml of distilled H_2O. The solution was magnetically stirred for 10 minutes at 40 °C and subsequently kept at 80 °C for several days. The obtained gel was annealed at 700 °C for 30 minutes. When the annealing process was completed, green powder was rinsed several times with distilled water. Finally, the excess of water was evaporated.

Another sample was obtained in the same way, with the only difference that sodium hydroxide (2.2 g of NaOH dissolved in a small amount of water) was poured into the stirring solution. The sample was further subjected to the identical procedure. After the annealing treatment, the obtained powder was green and its surface was covered by a thin layer of white powder. The washing process was repeated 4 times.

X-ray diffraction measurements

The diffraction patterns of the samples obtained with and without using NaOH are presented in Figure 1 and Figure 2, respectively.

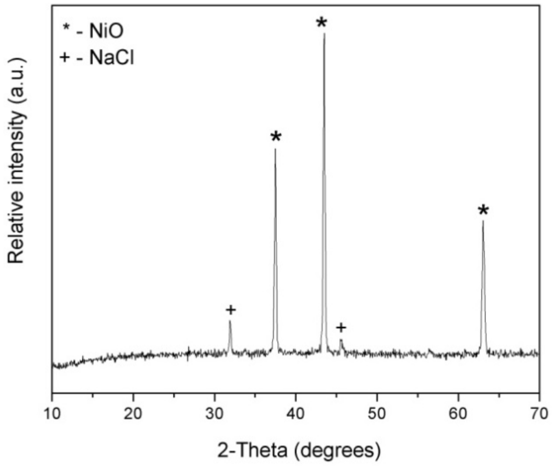

Figure 1 – XRD pattern of the sample prepared by using NaOH
Рис. 1 – Дифрактограмма образца, подготовленного при использовании NaOH
Слика 1 – Дифрактограм узорка припремљеног коришћењем NaOH

The sample presented in Fig.1 contained two phases, NiO (Qualx card No. 00-101-0093) and sodium chloride, NaCl (Qualx card No. 00-100-0041). Scherrer's formula was used to estimate the diameter crystallite size, d_{cr}. Both observed phases are nano-sized: d_{cr} (NiO, 2θ = 43.6°) = 34 nm, d_{cr} (NaCl, 2θ = 31.9°) = 36 nm, although the difference in the quantity and crystallinity of the observed phases resulted in significantly different intensity of the characteristic diffraction maxima.

Literature data proposed a mechanism for the preparation of NiO nanoparticles by using NaOH (Bahari Molla Mahaleh et al, 2008):

$$2NaOH_{(s)} \rightarrow 2Na^{+}{}_{(aq)} + 2OH^{-}{}_{(aq)}$$

$$NiCl_2 \cdot 6H_2O_{(s)} \rightarrow Ni^{2+}{}_{(aq)} + 2Cl^{-}{}_{(aq)} + 6H_2O_{(aq)}$$

$Ni(OH)_{2x} \cdot (H_2O)_{(s)} \rightarrow Ni(OH)_{2(s)} +_{x} H_2O_{(g)}$ (lower temperatures)

$Ni(OH)_{2(s)} \rightarrow NiO_{(s)} + H_2O_{(g)}$ (higher temperatures).

The absence of the usage of surfactant (polyvinilpyrolidone, polyethylene glycol, or cetyl trimethyl ammonium bromide) during the synthesis procedure resulted in the pronounced reaction:

$$2Na^{+}{}_{(aq)} + 2Cl^{-}{}_{(aq)} \rightarrow 2NaCl_{(aq)}\,.$$

When the solubility product of NaCl is overcome, a physical transformation

$NaCl_{(aq)} \rightarrow NaCl_{(s)}$ takes place, and NaCl arises as an impurity. The observed presence of NaCl pointed to the fact that the sample had been washed with an insufficient amount of water.

Figure 2 presents the sample obtained without NaOH. According to 2θ positions of the diffraction maxima, the sample contained a pure NiO phase of high crystallinity, d_{cr} (2θ = 43.6°) = 42 nm. The hkl planes corresponding to the positions of the diffraction maxima are shown in Fig. 2.

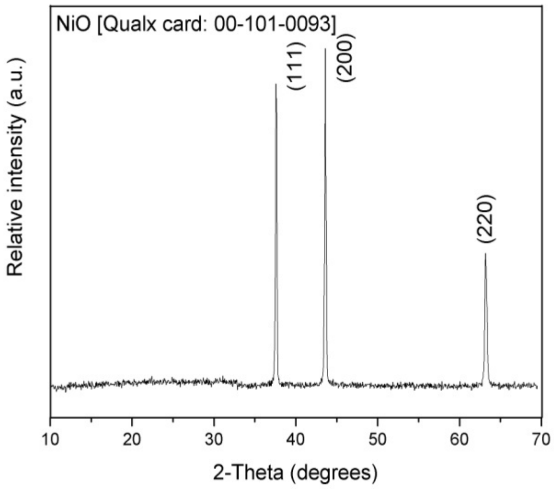

Figure 2 – XRD pattern of the sample prepared without using NaOH
Рис. 2 – Дифрактограмма образца, подготовленного без NaOH
Слика 2 – Дифрактограм узорка припремљеног без NaOH

If we recall the literature data, it can be noticed that Maia et al. obtained pure NiO by using NaOH in the synthesis, but performing annealing at 350 °C for different annealing times (9-12 hours) (Maia et al, 2006). On the other hand, they reported that the synthesis procedure without NaOH, followed by annealing at 700 °C for 30 minutes, resulted in the formation of bigger NiO nanoparticles, 62 - 78 nm (nearly two time bigger size than the size of the prepared NiO particles - Figure 2). The observed discrepancies between the experimental results could be explained in term of the differences in the composition of the used commercial gelatin.

Based on Figures 1 and 2, it can be concluded that the obtained NiO nanoparticles had a lot of similarities in size and structural properties (the diffraction maxima ascribed to the NiO phase in both samples appeared very similar). For this reason, the investigation of the properties of NiO nanoparticles continued with the sample containing pure NiO nanoparticles.

FTIR measurement

To investigate the surface of the prepared NiO nanoparticles, the FTIR spectrum was recorded in the mid-wave infrared region, between 2000 and 400 cm^{-1}. Figure 3 presents the spectrum in the range 1300 - 400 cm^{-1}, since in the region 1300 - 2000 cm^{-1} the curve is presented as a straight line.

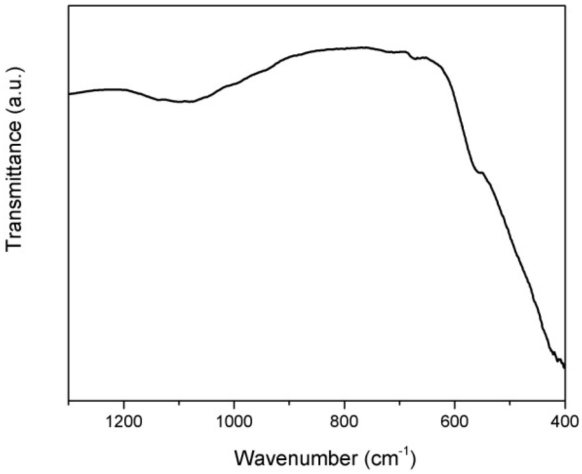

Figure 3 – FTIR spectrum of the sample prepared without using NaOH
Рис. 3 – FTIR спектра образца, подготовленного без NaOH
Слика 3 – FTIR спектар узорка припремљеног без NaOH

In the low-wavenumber part of the spectrum, there is a shoulder noticeable around 530 cm^{-1} and it can be ascribed to Ni - O vibration (Rahdar et al, 2015). A small peak at 640 cm^{-1} could be ascribed to stretching vibrations of the Ni - O - H bond (Rahdar et al, 2015). The observed vibrations confirmed the formation of NiO nanoparticles although the positions of both vibrations were shifted to higher wavenumber values, which is often a consequence of the presence of moisture in the sample. This is confirmed by the presence of a broad bond around 1080 cm^{-1}. Since one of the precursors in the synthesis was gelatin, and having in mind that C - O and C - C vibrations appear in the FTIR spectrum in the region 1090 - 1050 cm^{-1} (Faust, 1997), the observed bond points to the presence of gelatin carbon residues attached to the surface of NiO nanoparticles. It is important here to notice that the observed gelatin moisture is present after annealing at 700 °C, although Chuaynukul et al found that the degradation of gelatin occurred around 250 °C (Chuaynukul et al, 2014). This is a neither usual nor surprising result, since literature data reveal a well-known fact that substantial variations in the compositions of many commercial gelatins result in shifting gelatin degradation temperatures (Ling, 1978).

According to Figure 2 and Figure 3, it can be assumed that the investigated sample contained NiO nanoparticles, and that carbon residues were attached to the NiO nanoparticle surface.

Raman measurement

The Raman spectrum of the investigated sample in the spectral range from 100 to 1200 cm^{-1} is shown in Figure 4.

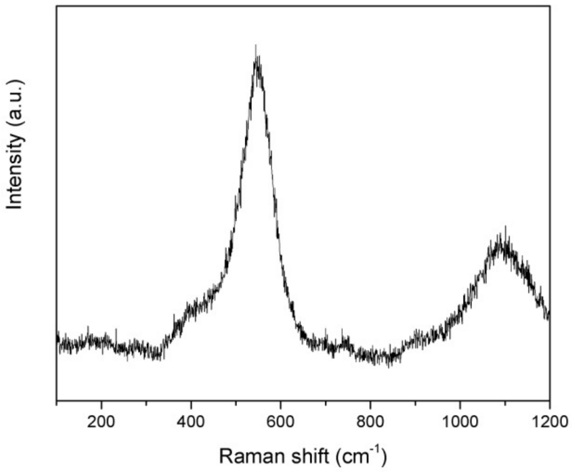

Figure 4 – Raman spectrum of the sample prepared without using NaOH
Рис. 4 – Рамановский спектр образца, подготовленного без NaOH
Слика 4 – Рамански спектар узорка припремљеног без NaO

It can be easily observed that Figure 4 is in accordance with Figure 3. The peak about 530 cm^{-1} points to the presence of NiO nanoparticles, and arises due to the Ni-O stretching mode (Cordoba-Torresi et al, 1991). The mode around 1085 cm^{-1} depicts the appearance of carbon moisture. The results obtained by using Raman spectroscopy confirmed the data observed by FTIR spectroscopy.

To understand the observed results, it is important to elucidate the mechanism of the formation of NiO nanoparticles by using gelatin in the synthesis procedure.

In literature, the chemical content of gelatin is often described as a mixture of hydroxyproline ($C_5H_9NO_3$), proline ($C_5H_9NO_2$) and glycine ($C_2H_5NO_2$), in different ratios (Jagadeesh et al, 2016). The sequence that describes gelatin, Gly-X-Y (where Gly refers to glycine, and X and Y are proline and hydroxyproline, respectively (Samouillan et al, 2011)), presents the triple helical structure of three left-handed polyproline type helices twisted into a superhelix. Thermal treatment initiated triple helix unfolds to produce random chains of gelatin (Harrington & von Hippel, 1969), that could be covalently linked to each other or not, depending on the annealing temperature (Privalov et al, 1979) (Balian & Bowes, 1977).

The proposed mechanism of the performed synthesis could be described in terms of the reaction between nickel salt and glycine:

$$NiCl_2 \cdot 6H_2O_{(s)} + C_2H_5NO_{2(s)} \rightarrow [NiCl_2(C_2H_5NO_2(H_2O)_2)]_{n(s)}$$

Fleck & Bohaty (Fleck & Bohaty, 2004) pointed out the main characteristic of this compound - containing helical chains of $[NiO_4Cl_2]$ octahedra connected by glycine molecules. It is noteworthy that literature data reveal that octahedral nickel-chloride complexes are characterized by a low value for the octahedral Ni-Cl bond order, which is caused by steric interactions between Cl^- and other ligands in the octahedral coordination sphere (See et al, 1998). Accordingly, the annealing process results in the release of chloride ions and their transformation to chloride gas. As a result, NiO nanoparticles containing surface organic impurities (originated from glycine molecules that connected $[NiO_4Cl_2]$ units) are obtained.

It is important to notice that gelatin could be obtained from vegatable origin (algae), or from animal origin (fish or bovine). Also, literature data confirmed that different manufacturers sometimes add formaldehyde or glutaraldehyde in the gelatin preparation in order to ensure hardening of gelatin (Chiellini et al, 2001). Since the chemical composition of gelatin varies dependent on the origin of gelatin, as well as on the manufacturing procedure, the purity of NiO nanoparticles could be improved by varying the synthesis parameters, which could be done by two different approaches: to change the type of gelatin, or to use gelatin manufactured by different producers.

SQUID measurement

Although SQUID measurements allowed the investigation of the magnetic properties of the sample, this type of examination is important for the estimation of the suitability of NiO nanoparticles for gas sensing application due to the coupling between the electric and magnetic fields in the NiO nanoparticle system. Since NiO nanoparticles showed different magnetic properties dependent on the type of synthesis and their structural properties, a significant deviation from the desired magnetic properties could be ascribed to the alteration of NiO electronic properties.

The hysteretic loops of the sample are shown in Figure 5.

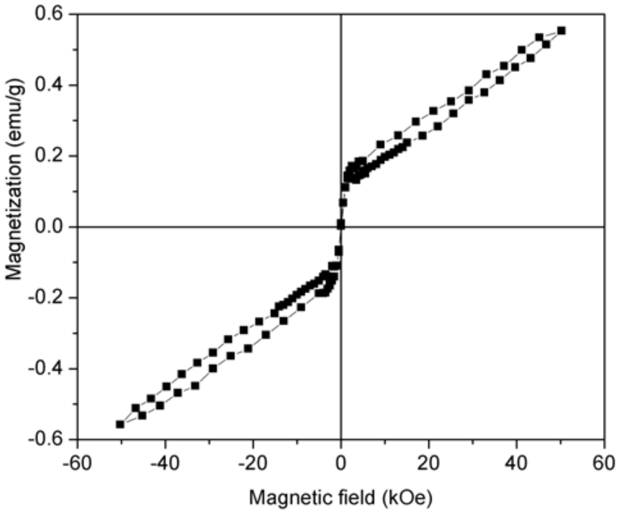

Figure 5 – M(H) curves of the sample prepared without using NaOH
Рис. 5 – M(H) кривые образца, подготовленного без NaOH
Слика 5 – M(H) криве узорка припремљеног без NaOH

Recording the hysteretic curves enabled the estimation of the intrinsic coercivity field value (H_{ci} = 65 Oe) and saturation magnetization (M_s = 0.55 Oe). The obtained Ms value is expected for NiO nanoparticles (Taghizadeh, 2016). It is shown in literature that although bulk coercivity of NiO is around 0.8 Oe, NiO nanoparticles showed increased averaged coercivity due to size effects (around 49 Oe) (Taghizadeh, 2016). Accordingly, the coercivity observed in Figure 5 is characteristic for NiO nanoparticles.

It is noteworthy that the measured hysteretic loops possess constricted middles, so-called "wasp-waisted" curves (Tauxe et al, 1996), that are often oversimplifyingly interpreted, which could result in erroneous conclusions. This shape of the curves could be a consequence of the presence of other phases in the samples. If we consider Figure 1, the presence of other phases is unlikely. Another reason of the modified shape of the hysteretic curves could be the presence of the surface moisture reflecting in the lowered saturation magnetization value and the modification of other hysteretic parameters (Durmus et al, 2011), (Lu et al, 2002). Since the investigated sample showed the M_s and H_{ci} values characteristic for NiO nanoparticles, it is clear that the observed feature could not appear for this reason. For a more precise interpretation of the magnetic properties of the investigated sample, in-depth magnetic investigation consisting of the application of different measurement protocols and a comprehensive interpretation of the obtained results should be carried out.

Conclusion

This article presents a preliminary investigation of the properties of NiO nanoparticles that could be used for the preparation of sensors for H_2S gas detection. Two samples are obtained by a combination of the coprecipitation method and the annealing treatment at 700 °C for 30 minutes. XRD measurements of the samples have revealed that the presence of NaOH in the synthesis route influences the purity of the samples. The samples obtained by using NaOH contained moisture - NaCl; the observed moisture could be eliminated by washing the sample with a significant amount of water. The sample prepared without NaOH contained pure NiO nanoparticles. FTIR and Raman spectroscopy have confirmed the presence of carbon moisture in the form of C - C or C - O bonds, attached to the surface of NiO nanoparticles. The noticed obstacle could be overcome by using commercial gelatin of some other producer, which would experience thermal degradation at lower temperatures. Hysteretic measurements have revealed the presence of wasp-wasted curves although the Ms (0.55 emu/g) and Hci (65 Oe) values are characteristic for NiO nanoparticles. Further investigation will be performed in the future.

Notations and symbols

ATR - attenuated total reflection
d_{cr} - crystallite diameter
FTIR - Fourier transform infrared spectroscopy
hkl - Miller indices of the plane
H_{ci} - intrinsic coercivity field
M_s - saturation magnetization
SQUID - superconducting quantum intereference device
XRD - X-ray diffraction
λ - Cu Kα radiation wavelength
θ - Bragg angle

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ХАРАКТЕРИСТИКИ НАНОЧАСТИЦ NiO, ПОДГОТОВЛЕННЫХ С ПОМОЩЬЮ ЖЕЛАТИНА ПРИ ПРОСТОМ И НЕДОРОГОМ МЕТОДЕ ОБОБЩЕНИЯ

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Резюме:

Нанокомпозитные материалы, содержащие наночастицы NiO, нанесенные на нанопровода WO_3 можно использовать для мониторинга уровня H_2S-газа. Эффективность данного материала в основном определяется свойствами этапа NiO. Проведенное исследование представляет собой первый этап в подготовке модифицированного материала NiO/WO_3. В данной статье представлено получение наночастиц NiO простым и недорогим методом обобщения. Поведение наноразмерных частиц NiO исследуется с помощью рентгеновской дифракции, инфракрасной спектроскопии с Фурье-преобразованием, рамановской спектроскопии и гистерезисных измерений.

Ключевые слова: NiO, дифракционные измерения, спектроскопия, наночастицы, материалы.

КАРАКТЕРИЗАЦИЈА NiO НАНОЧЕСТИЦА ПРИПРЕМЉЕНИХ ПОМОЋУ ЖЕЛАТИНА ТОКОМ ПОСТУПКА „LOW-COST” СИНТЕЗЕ

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ЈЕЗИК ЧЛАНКА: енглески

Сажетак:

Нанокомпозитни материјал који садржи наночестице NiO нанете на WO_3 наножице може се користити за мониторинг нивоа H_2S гаса. Ефикасност овог материјала највећим је делом одређена својствима NiO фазе. Изведена студија представља прву фазу у припреми модификованог материјала NiO/WO_3. У овом раду NiO наночестице су припремљене једноставним „low-cost” методом синтезе. Понашање наноструктурних NiO честица испитивано је рендгенском дифракцијом, инфрацрвеном спектроскопијом са Фуријеовом трансформацијом, раманском спектроскопијом и хистерезним мерењем.

Кључне речи: NiO, дифракциона мерења, спектроскопија, наночестице, материјали.

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