INVESTIGATION OF SEMICONDUCTOR OXIDE NANOPARTICLE AND MILLED MWCNT-BASED THIN FILMS AND THEIR SUBSTRATES

PH. D. THESIS

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1. Introduction and objectives

Semi-conducting metal oxides, carbon nanostructures, and conductive polymers are materials that are gaining increasing attention in the field of electronics and sensing owing to their unique properties and potential applications. Zinc oxide is a wide bandgap semiconductor with special conducting and optical properties. Multi-walled carbon nanotubes provide high mechanical strength and excellent electrical conductivity, while PEDOT:PSS is a widely used polymer in organic-inorganic-based composite layers. All three materials can be used in a variety of electronic applications such as solar cells, sensors, and transistors. The combination of these materials can result in high electron mobility, outstanding mechanical properties, and good electrical conductivity. The application of these sensors containing semiconducting oxides, nanotubes, and conductive polymers in proper composites is increasing. Although the use of nanosensors is becoming more widespread and the global market of the industry is growing rapidly, there are still many opportunities for development.

Our research's main focus was examining various nanoparticles and substrates. Our goal is also to produce and study a possible sensor layer using the aforementioned materials. In the early stages of the work, our goal was to produce size-controlled multi-walled carbon nanotubes using a planetary ball mill and to optimize the milling parameters. Carbon nanotubes are widely used 1D nanostructures, but the advantageous length-to-diameter ratio can be a disadvantage in certain cases, such as leading to aggregation during the preparation of composite films, which can be reduced by controlling the MWCNT length through milling. Additionally, our aim was to find an explanation of how the mechanical treatment affected the morphology and nitrogen adsorption behavior. We also wanted to investigate how the thermodynamic and dielectric properties of the later-produced thin-layer substrates change as a result of a coating containing nanoparticles. To achieve this, a ZnO nanorod layer was prepared, and as a reference, we examined the amorphous PET and ITO-coated PET substrates using TGA, MDSC, DMA, and DRS methods.

During this work, the goal was to create a thin film on a transparent and flexible polymer substrate that can also function as a sensor by combining the advantageous properties of the previously mentioned materials. In the production of the thin layer, ZnO nanorods, carbon nanotubes, and PEDOT:PSS was used. The aim was to provide a possible alternative to the ITO thin layer, which, although has many positive properties, is relatively expensive, has a limited supply, and due to its fragility has low resistance against mechanical effects. After examining the components of the film and the substrates separately, the composite thin layers produced on different carriers were investigated. The aim was to produce a thin film having relatively high transparency which is able to function as a UV and ethanol sensor, working at room temperature, without heating the test chamber.

2. Experimental section

In the first step, the nanoparticles for further milling experiments, and for the preparation of thin films were synthesized. The multi-walled carbon nanotubes were produced by the CVD technique, using acetylene gas as the carbon source in the presence of Fe-Co/MgO (2.5% mass% metal: carrier ratio) catalyst. The synthesis was carried out in a quartz reactor under an inert atmosphere. The milling of the carbon nanotubes was carried out using a Fritsch Pulversiette 6 (FP6) type planetary ball mill, in which the milling parameters can be accurately set. In total, five milling series were performed, during which several settings were varied, but only one parameter was changed per series: the milling speed from 150 to 600 rpm, the milling time from 5 to 60 minutes, the number of 10 mm grinding balls from 5 to 25, the number of 5 mm diameter balls from 40 to 200, and the mass ratio of larger and smaller balls from 10:1 to 1:10.

The ZnO nanoparticles were produced by a solvothermal method, using zinc acetate (c=0.2 M) as a precursor. In methanol medium, as a first step, NaOH (c=0.4 M) solution was added dropwise to the zinc acetate solution forming a white precipitate ($Zn(OH)_2$) instantly, followed by a heat treatment: for 6 hours in a Teflon-lined autoclave at 428 K. During the production of thin layers, a ZnO nanoparticle coating on APET or ITO-coated PET surfaces was prepared to investigate the changes in thermomechanical and dielectric properties. After creating seeds of ZnO nanoparticles by mixing zinc acetate in methanol at a concentration of 0.03 mol/dm^3 with NaOH and treating at 333 K for 2 hours, using a spin coater, a uniform thin layer of ZnO seeds was prepared on the surface of the APET or ITO/PET. After drying the uniform nanoparticle coating, nanorods were grown from the ZnO seeds using zinc acetate and diethylene triamine.

In order to perform sensor tests, a thin film with suitable conductivity and transparency was needed. To produce the most appropriate layer, several coatings were prepared: only ZnO, only MWCNTs, only PEDOT:PSS, MWCNT/PEDOT:PSS, and ZnO/MWCNT/PEDOT:PSS composites on APET and ITO coated PET surfaces.

The morphology and structure of the prepared and modified nanoparticles were examined using a FEI Tecnai G2 20 X-Twin type transmission electron microscope with a 200 kV accelerating voltage. During sample preparation, the ethanolic suspension of the sample was dried to a carbon film-coated copper grid (300 mesh). Three important data were obtained from the nitrogen adsorption isotherms of the milled carbon nanotube samples, at

77 K: the specific surface area (A_s, BET), which carries information about the total surface, the pore size distribution (PSD), which characterizes the porosity of the material, and the fractal dimension of the surface (D_S), which gives information about the uniformity of the surface.

During the light scattering measurements, a Zetasizer ZS device was used to measure the polydispersity index and Zeta potential of the selected nanotube samples in quartz cuvettes, which can provide information on how the measured nanoparticles behave in dispersions of certain solvents. The morphology of the produced coatings/films was examined using a Hitachi S-4700 scanning electron microscope, using a 10 kV acceleration voltage and a maximum resolution of 1.5 nm. XRD spectra of ITO/PET and ZnO nanoparticle-coated samples were measured in the $2\Theta = 3\text{-}60°$ angle range using a Rigaku Miniflex II device, using Cu Kα ($\lambda = 1.5418$ Å) as the radiation source. The transmittance of the thin layers and substrates was studied using a portable Ocean Optics UV-Vis spectrometer, utilizing a USB-4000 type detector and a DH-2000-BAL light source (deuterium and halogen lamps).

During thermogravimetric measurements, the polymer samples and coated polymer substrates were examined using a TA Instruments Q500 TGA device in inert nitrogen and oxidative air flow, in the range of 298-773 K, using a heating rate of 5 K/min. The DSC measurements were performed using a TA Instruments Q200 device in modulated mode (0.5 K/min) with Tzero aluminum sample holders at a 3 K/min heating and cooling rate. The viscoelastic properties of the polymer substrate samples were studied by dynamic mechanical analysis. The measurements were performed in oscillation mode, using a DMA Q800 device. In this mode, the sample gives a sinusoidal response to the sinusoidal mechanical effect applied to it, and the phase shift between them is informative. The storage and loss moduli and the tan δ curve were detected and analyzed. For polymer substrate samples, isochronal DRS measurements were performed at 1 K/min cooling and the same heating rate in the frequency range of 10^{-1} –10^{6} Hz. The dielectric spectrum was recorded using a Novacontrol Alpha-A unit.

During the sensor measurements, a LabJack UE9 control unit, Keithley A Tektronix Company 2401 flow meter, AALBORG - GFC17 type flow regulator, and AALBORG controller were used at room temperature (298 K) and atmospheric pressure. First, the response of the ZnO/MWCNT/PEDOT:PSS film deposited on the APET substrate to UV excitation was measured. Then, for 90 seconds, ethanol was allowed to flow into the test chamber, and after regeneration, the exposure was repeated several times to get information on repeatability. In the second measurement series, the ethanol concentration was changed in cycles, different concentration of the target was allowed into the sample chamber with the same exposure and regeneration time.

3. Novel scientific results:

T1: Using a defined energy milling, size-controlled carbon nanotubes were produced and examined by electron microscopy and N_2 adsorption measurements.

1.1. We analyzed the TEM images of carbon nanotube samples produced in five measurement series using the Image Java program to interpret the controlled size changes that occurred due to mechanical effects. Based on the resulting histograms, we determined the optimal parameters for the following sample preparation. The average nanotube lengths obtained under different milling parameters were plotted and we could verify that the steep break which is observable in the initial stages of milling is influenced by two studied values: the milling energy and the number of impacts. Based on this, we concluded that the determination of the number and size of the milling balls is a much more important factor in the production of size-controlled nanotubes than the milling time, and thus with proper design, a shorter time (and thus less energy invested) may be sufficient to achieve the desired nanotube length.

1.2. From the nitrogen adsorption-desorption isotherms recorded at 77 K we were the first to determine three important numerical descriptors on the morphology of the milled carbon nanotubes simultaneously in the literature: the specific surface area (A_s, BET), the pore size distribution (PSD), and the surface fractal dimension (D_S), which provides information about the uniformity of the surface. We showed that in the case of the multi-walled carbon nanotubes, the non-integer D_S values obtained after mechanical treatment are the result of deviations from the ideal cylinder surface, which can be caused by various local defects, holes, or amorphous carbon deposits. The ends of the tubes are closed by a half fullerene-like "cap" before milling, without mechanical treatment, the inner pores of the tubes were inaccessible to the adsorbed molecules. The specific surface area and the surface fractal dimension always increased with milling, showing a characteristic maximum. We have shown that the material underwent significant morphological changes due to mechanical treatment: based on TEM images and N_2 adsorption isotherms, we assumed that the new PSD maximum is formed because the opening of the nanotube interior creates new adsorption sites. The obtained results confirmed our hypothesis that the opening of the tube dominates over the amortization of the nanotube wall.

T2: ZnO nanorods were produced on amorphous PET and different layer resistance ITO/PET substrates.

2.1. We have developed a method for producing ZnO nanorods directly on the surface of PET or ITO-coated PET substrates. First, ZnO nanorod "seeds" were created using zinc acetate precursor. The ZnO nanorods were then applied to the surface of the PET and ITO/PET using a spin coater, repeating the "seed" layer application several times to create a uniform thin layer. Finally, ZnO nanorods were grown from the ZnO "seeds" by treating the substrate containing the thin layer with a solution of zinc acetate and diethylene triamine at a specific temperature. The uniform morphology of the ZnO nanorod coating was confirmed by SEM images and XRD measurements. The effect of the ZnO nanorod coating on transparency was characterized by UV-Vis transmittance measurements.

2.2. We were able to interpret the one-step thermal degradation characteristic of PET observed during TG analysis performed in an inert nitrogen atmosphere in the case of substrates (PET, ITO/PET) and ZnO nanorod-coated ITO/PET. It was shown that in an oxidative environment, air atmosphere, the amorphous PET substrate still exhibited one-step thermal degradation, but at a lower temperature. It was also shown that in an oxidative environment, the ITO/PET and ZnO nanorod/ITO/PET samples underwent thermal degradation in two steps, and the effect of ZnO on the degradation temperature was observed.

2.3. Based on the results of DSC measurements, we showed the amorphous PET sample case, the well-defined enthalpy relaxation-induced glass transition (Tg) appears in all heating cycles as an overlapping endothermic peak. The melting and crystallization processes were observed. In the third cycle, the T_g is followed by a partial transition, the cold crystallization process, a thermally induced crystallization, which occurs when the polymer is heated above the glass transition and not cooled quickly enough. We showed that this transition was only observed in the case of the amorphous PET sample, the reason for this is probably the different degree of crystallinity.

T3: The thermomechanical and dielectric relaxation properties of ZnO nanorod-coated amorphous PET and various layer-resistance ITO/PET substrates

3.1. We were able to interpret the observed differences in the thermomechanical behavior of ZnO nanorod-coated amorphous PET and ITO/PET substrates. We obtained information about the viscoelastic properties of our material from oscillation DMA measurements. In our case, we used the peak of the tan δ and the peak of the loss modulus to determine the glass transition temperatures under different frequency excitations. We could observe that with higher measurement frequencies, the tan delta and loss modulus (E") peak temperatures (alpha-relaxation) increased and the tan delta peak widened. We confirmed that the amorphous PET showed different behavior, the glass transition occurred at a lower temperature than in the case of ITO-coated PET samples. We also observed differences among the various layer-resistance ITO/PET samples due to the different ITO-PET ratios, the T_g was higher in the case of the samples with higher layer resistance. In every case, the ZnO nanorod coating caused an increase in the glass transition temperature This phenomenon was proven to be independent of the frequency applied and the ITO/PET nominal resistance as well.

3.2. The observed differences in the dielectric relaxation behavior of ZnO nanorod-coated amorphous PET and ITO/PET substrates were interpreted. To investigate the molecular dynamics of the PET phase in ITO/PET-based samples, we used DRS, following the frequency dependence of the relevant dielectric relaxations. Using the isochronal curves obtained, we interpreted the temperature dependence of the α-relaxations and secondary β-relaxations that provide information on the segmental dynamics using Arrhenius diagrams. We determined the E_a activation energies and calculated the T_v Vogel temperatures from the α-relaxations. Based on the calculated values, we showed that the uncoated PET has a lower activation energy than the ITO-coated. The higher activation energies and lower T_v values also confirmed the cold crystallization process which was observed during DSC measurements. For the ITO-coated PET samples, we assumed a higher degree of crystalline order based on the Vogel temperatures calculated from the α-relaxations. The ZnO nanorod coating decreased the activation energies and increased the Vogel temperatures compared to the amorphous PET substrate and the measured values were closer to the ITO/PET substrate. We also showed the change in dynamic fragility factor in the case of samples coated with ZnO nanorods.

T4: The sensor capability of the ZnO/MWCNT/PEDOT:PSS thin film produced on a flexible substrate

4.1. We proved that the ZnO/MWCNT/PEDOT:PSS film gives a reversible photoelectric response under UV illumination. We proposed the following theory to explain the phenomenon. Reduced conductivity was observed in the dark, which was caused by the adsorption of oxygen molecules on the surface of the n-type semiconductor ZnO nanoparticles, which removed free electrons and increased the depletion layer, thus increasing the resistance. A decrement in the resistance was observable when the UV light source was turned on, which was attributed to the formation of new electron-hole pairs caused by the absorption of photons, and the recombination of negative oxygen ions with the newly formed holes which reduced the depletion layer, thus increasing conductivity. This process may be aided by the presence of p-type semiconductor MWCNT and hole-transporting conductive polymer in the layer.

4.2. We proved that the ZnO/MWCNT/PEDOT:PSS thin layer applied to the surface of the APET substrate shows a linear sensitivity to ethanol in the tested concentration range, and that the measurements are reproducible with a short regeneration time. A decrement was observed in conductivity when ethanol was introduced to the test chamber, which was attributed to the p-type characteristic of CNT and the possible formation of a p-n heterojunction between ZnO nanoparticles and multiwalled carbon nanotubes, where the PEDOT:PSS may have helped the conductivity as a flexible hole-type semiconductor matrix. Since in the case of p-type semiconductors the conductive behavior is dominated by positive charge carriers, and holes, therefore, the addition of a typical hole transporter, a conductive polymer, could result in a faster response due to percolation effects.

4. Practical environmental implications of the results

We concluded from the milling tests of carbon nanotubes that determining the number and size of the milling balls is a much more important factor in producing size-controlled nanotubes than the milling time, so with proper design, a shorter time (and thus less energy input) is sufficient to achieve the desired nanotube length. Based on this consideration, with appropriate experimental design, and choosing the optimal parameters, the desired size of the nanostructured material can be reduced in a shorter time and thus with less energy consumption.

We also concluded from the examination of samples produced on the surface of different substrates, that in certain cases, the appropriate composite thin film can replace the usage of the expensive and limited ITO. In the case of only semiconductor metal oxide-based sensors, a high working temperature of 373-723 K is required. The operating temperature of sensors can be reduced by forming a hybrid thin film, thus reducing the energy required to heat the test chamber. For the operation of an ethanol sensor containing pure ZnO nanoparticles, it is essential to heat it to a temperature of 473-573 K, for which a heating panel with a power of at least 100 mW- 1 W is required, in contrast, the composite layer we tested was able to work at room temperature. Furthermore, producing various electronics and sensors on a flexible substrate points towards miniaturization and reduction of energy consumption.

Although sunlight is one of the main environmental resources that keeps all the organisms alive on earth, and UV radiation is essential for vitamin D synthesis and helps to reduce the risk of some diseases, excessive UV exposure can lead to adverse effects, including eye diseases, premature aging, sunburn, and skin cancers. The aforementioned reasons make UV monitoring essential.

The concentration of ethanol is an important parameter in many areas: detecting emissions from industrial sources, controlling fermentation processes in the production of biofuels, testing food packaging materials, or even in the analysis of exhaled air.

The sensor layer on the flexible substrate we tested was able to work as a UV and ethanol detector, works at room temperature, and shows acceptable transparency.

5. Publications related to the thesis:

1. **Effect of planetary ball milling process parameters on the nitrogen adsorption properties of multiwall carbon nanotubes**

   Ibolya Zita Papp, Gábor Kozma, Róbert Puskás, Tímea Simon, Zoltán Kónya, Ákos Kukovecz

   Adsorption, 2013, 19 (2-4)

   DOI: 10.1007/s10450-013-9493-8

   IF 2013/2014: 1.735 Independent citations:8

2. **Investigation into the effect of ZnO nanorod coating on the thermal-mechanical and dielectric properties of ITO coated PET**

   Ibolya Zita Papp, Angel Alegría, Zoltán Kónya, Ákos Kukovecz

   Materials Research Bulletin, 2021, 149(11):111701,

   DOI: 10.1016/j.materresbull.2021.111701

   IF 2021/2022: 5.6 Independent citations:3

3. **Fabrication and characterization of a bifunctional zinc oxide/multiwalled carbon nanotube/ poly(3,4-ethylenedioxythiophene): polystyrene sulfonate composite thin film**

   Ibolya Zita Papp, Adél Szerlauth, Tímea Szűcs, Péter Bélteky, Juan Fernando Gomez Perez, Zoltán Kónya, Ákos Kukovecz

   Thin Solid Films, 2023, 778:139908

   DOI: 10.1016/j.tsf.2023.139908

   IF 2021/2022: 2.1 Independent citations:-

6. Conference contributions:

1. Ball milling experiments on multiwalled carbon nanotubes

Ibolya Zita Papp, Gábor Kozma, Zoltán Kónya, Ákos Kukovecz

NAPEP (Nanotechnology Platform for Electronics and Photonics), 2012. Szeged (előadás)

2. Investigation of adsorption properties on multiwalled carbon nanotubes with different size milled by planetary ball mill

Ibolya Zita Papp, Gábor Kozma, Zoltán Kónya, Ákos Kukovecz

ISSHAC8 (Eighth International Symposium, Effects of Surface Heterogeneity in Adsorption and Catalysis on Solids), 2012. augusztus 27-31, Krakkó, Lengyelország (előadás)

3. Investigation of multiwalled carbon nanotubes modified cellulose-nitrate membrane films

Ibolya Zita Papp, Gábor Kozma, Zoltán Kónya, Ákos Kukovecz

Magyar Mikroszkópos Konferencia (HSM), 2012. május 10-12, Siófok (előadás)

4. Többfalú szén nanocsővel módosított membránok előállítása és vizsgálata

Papp Ibolya Zita, Kónya Zoltán, Kukovecz Ákos

Tudományos Diákköri Konferencia, 2012. április 26, Szeged (előadás)

5. Többfalú szén nanocsővel módosított membránok előállítása és vizsgálata

Papp Ibolya Zita, Kónya Zoltán, Kukovecz Ákos

Országos Tudományos Diákköri Konferencia, 2013. április 6, Eger (előadás)

6. Preparation of zinc-oxide nanoparticles modified surfaces and the investigation of its photocatalytic properties

Ibolya Zita Papp, Gábor Kozma, Zoltán Kónya, Ákos Kukovecz

Magyar Mikroszkópos Konferencia (HSM), 2013. május, Siófok (előadás)

7. Cink-oxid nanorészecskékkel bevont üvegfelületek előállítása és jellemzése

Papp Ibolya Zita, Kővári Dániel, Kukovecz Ákos

II. Környezetkémiai Szimpózium,: 2013. október 11., Dobogókő (előadás)

8. ZnO nanorészecskékkel módosított üvegfelületek előállítása és jellemzése

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9. Nano- és nagyszemcsés cink-oxid ökotoxikológiai hatása két eltérő táplálkozásmódú és életmenetű szabadon élő fonálféreg fajra

Hrács, Krisztina; Papp, Ibolya Zita; Kukovecz, Ákos; Brezina, Brigitta; Wilk, Tímea; Nagy, Péter

X. Magyar Ökológus Kongresszus, Veszprém, 2015

10. Removal of Acid Red 1 by TiO_2 nanoparticles coated polyethersulfone membrane under UV irradiation

Ildikó, Kovács; Szabolcs, Kertész; Sándor, Beszédes; Cecilia, Hodúr; Ibolya, Zita Papp; Ákos, Kukovecz; Zsuzsanna, László

Ozone and Advanced Oxidation Leading-edge science and technologies Proceedings: 22nd World Congress & Exibition, International Ozone Association (2015) Barcelona, Spanyolország : pp. 10.2-1-10.2-6. , 6 p.

Unrelated publications:

1. Experimental validation of the Burgio-Rojac model of planetary ball milling by the length control of multiwall carbon nanotubes

Kozma, G; Puskas, R; Papp, IZ; Belteky, P; Konya, Z; Kukovecz, A
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IF (2016/2017): 6.337 Independent citations: 3

2. Membrane fouling control by means of TiO2 coating during model dairy wastewater filtration

Ildikó, Kovács; Szabolcs, Kertész; Gábor, Veréb; Ibolya, Zita Papp; Ákos, Kukovecz; Cecilia, Hodúr; Zsuzsanna, László
Desalination and water treatment 73 pp. 415-421. , 7 p. (2017)
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3. Investigation of titanium-dioxide coatings on membrane filtration properties

Kovács, I; Beszédes, S; Kertész, S; Veréb, G; Hodúr, C; Papp, I Z; Kukovecz, Á; László,
Studia Universitatis Babes-Bolyai Chemia 62 : 1 pp. 249-259. , 11 p. (2017)
IF(2017/2018): 0.33 Independent citations: 10

4. Toxicity and uptake of nanoparticulate and bulk ZnO in nematodes with different life strategies

Hracs, Krisztina; Savoly, Zoltan; Seres, Aniko; Kiss, Lola Virag; Papp, Ibolya Zita; Kukovecz, Akos; Zaray, Gyula; Nagy, Peter
Ecotoxicology 27: 8 pp. 1058-1068. , 11 p. (2018)
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5. Production of meloxicam suspension using pulsed laser ablation in liquid (PLAL) technique

Béla, Hopp; Eszter, Nagy; Franciska, Peták; Tomi, Smausz; Judit, Kopniczky; Csaba, Tápai; Judit, Buda; Ibolya, Zita Papp; Ákos, Kukovecz; Rita, Ambrus et al.
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6. Membrane Association Modes of Natural Anticancer Peptides: Mechanistic Details on Helicity, Orientation, and Surface Coverage

Quemé-peña, M.; Juhász, T. ; Kohut, G.; Ricci, M.; Singh, P.; Szigyártó, I.Cs.; Papp, Z.I. ; Fülöp, L.; Beke-Somfai, T.

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Lima, B.; Ricci, M.; Garro, A.; Juhász, T.; Szigyártó, I.C.; Papp, Z.I.; Feresin, G.; Garcia, de la Torre J.; Lopez, Cascales J.; Fülöp, L.et al.

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IF (2022): 4.019 Independent citations: 7

8. The interaction of half-sandwich (η5-Cp*)Rh(III) cation with histidine containing peptides and their ternary species with (N, N) bidentate ligands

Hassoon, Azza A.; Szorcsik, Attila; Bogár, Ferenc; Papp, Ibolya Zita; Fülöp, Lívia; Kele, Zoltán; Gajda, Tamás

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9. Peptide-based chemical models for lytic polysaccharide monooxygenases

Hassoon, Azza A.; Szorcsik, Attila; Fülöp, Lívia; Papp, Zita I.; May, Nóra V.; Gajda, Tamás

Dalton Transactions 51: 45 pp. 17241-17254. , 14 p. (2022)

IF(2022): 4.569 Independent citations: -

SCIENTOMETRIC DATA

Sum of peer-reviewed publications: 12 In relation to the thesis: 3

Cumulative impact factor: 41,1 In relation to the thesis: 9,435

Sum of independent citations: 49 In relation to the thesis: 11