



NATIONAL INSTITUTE FOR RESEARCH AND DEVELOPMENT
IN ELECTRICAL ENGINEERING ICPE-CA

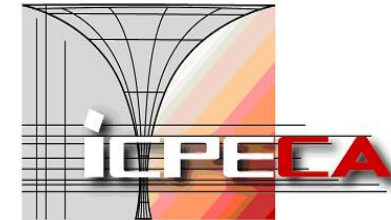


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NANOSTRUCTURED MATERIALS WITH SILVER NANOPARTICLES FOR ANTIMICROBIAL, SELF-CLEANING AND ELECTRICAL APPLICATIONS

**Dr. Chem. S. Gavriliu, Prof. Dr. W. Kappel, Dr. Eng. E. Enescu,
Dr. Eng. M. Lungu, Chem. A. Petica, Eng. F. Grigore**

***Seminar: "Building on the experience of European cooperation in nanoscience
and nanotechnologies", February 3rd, 2010, Romanian Academy***

NANOSTRUCTURED MATERIALS

FOR ANTIMICROBIAL AND SELF-CLEANING APPLICATIONS

- ❖ Colloidal silver solutions
- ❖ Photocatalytic and biocidale nanocomposite powders and slimes
- ❖ Silver nanopowders
- ❖ Acryl-styrene polymer dye with silver/titania nanocomposite powders
- ❖ Antibacterial and antifungal products doped with silver nanoparticles
- ❖ Hydrogel with silver nanoparticles

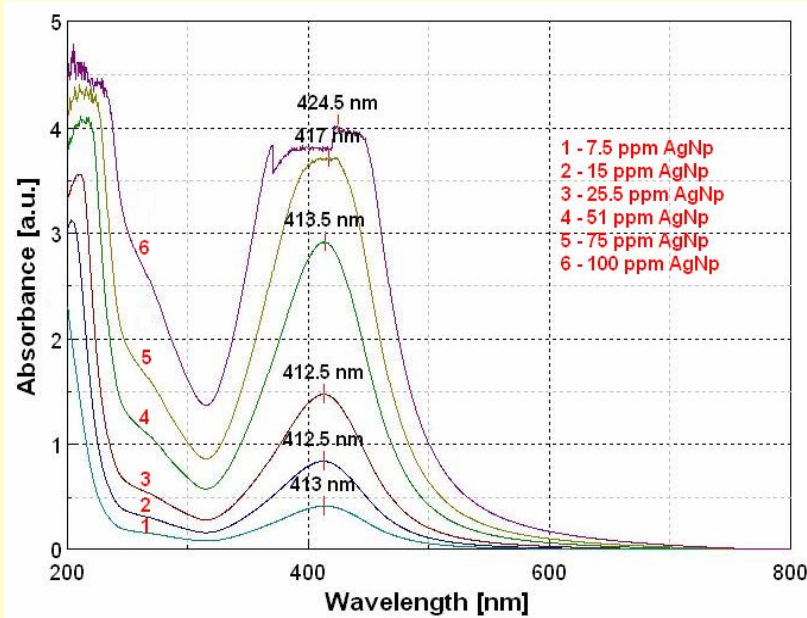
CHEMICAL COLLOIDAL SILVER SOLUTIONS

GENERAL DESCRIPTION: Ecological antimicrobial material in form of stable suspensions (CSS) with different concentrations of bioactive silver nanoparticles (NpAg), dispersed and stabilized in aqueous mediums.

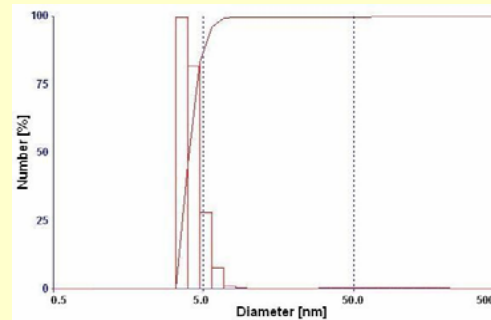


TECHNICAL CHARACTERISTICS:

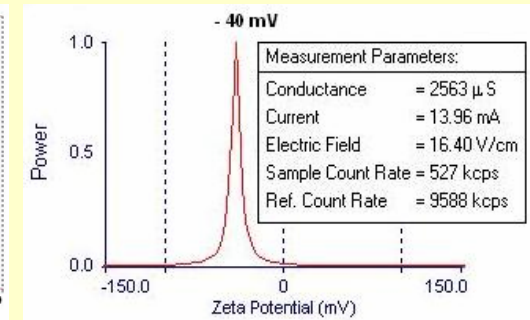
- NpAg concentration: 5...5.000 ppm
- average diameter of NpAg: 10 nm
- high stability of solution (zeta potential: -45...-33 mV)
- high oligodynamic, regenerative and antimicrobial activity



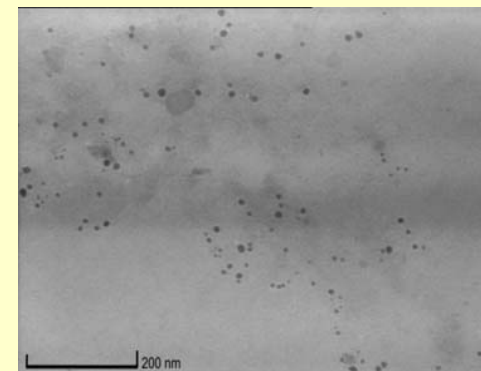
UV-Vis absorption spectra of CSS



Grain size distribution of NpAg



Zeta potential of a CSS



TEM image of NpAg

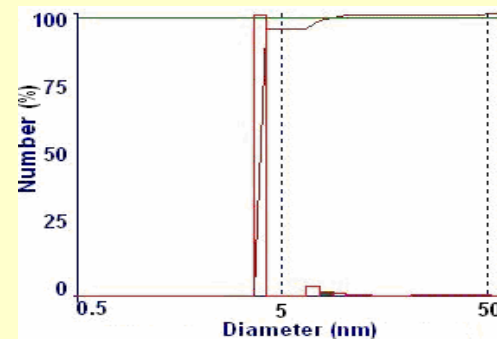
ELECTROCHEMICAL COLLOIDAL SILVER SOLUTIONS

GENERAL DESCRIPTION: Ecological antimicrobial material in form of stable suspensions (CSS) with different concentrations of bioactive silver nanoparticles (NpAg), dispersed and stabilized in aqueous mediums.

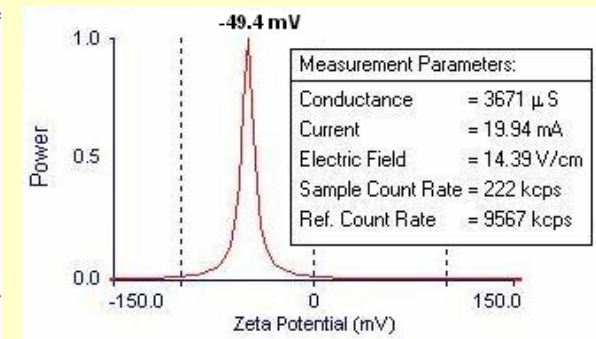


TECHNICAL CHARACTERISTICS:

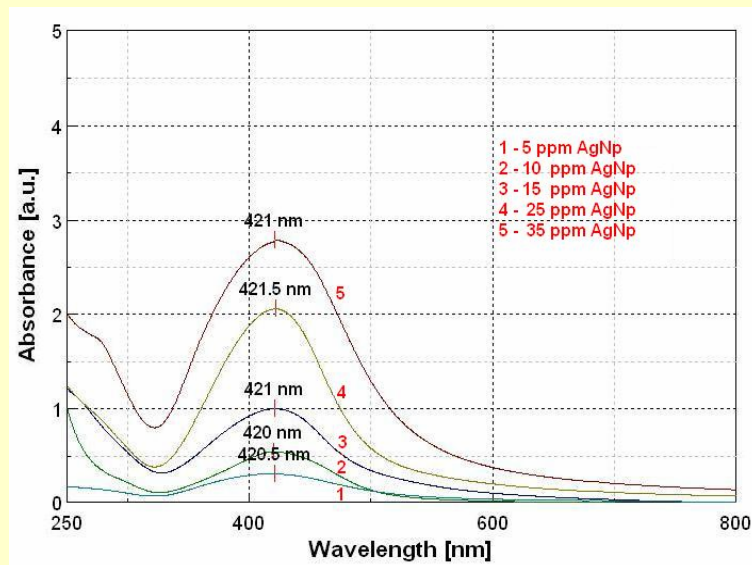
- NpAg concentration: 5...35 ppm
- average diameter of NpAg: max. 20 nm
- high stability of solution (zeta potential: -55...-35 mV)
- high oligodynamic, regenerative and antimicrobial activity



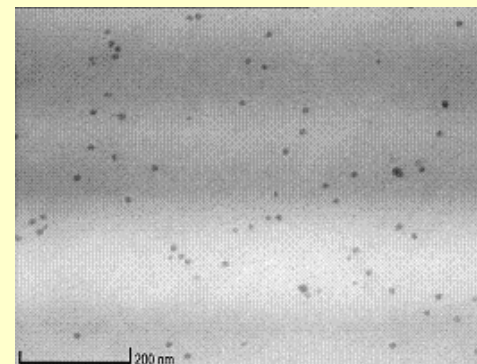
Grain size distribution of NpAg



Zeta potential of a CSS



UV-Vis absorption spectra of CSS



TEM image of NpAg

APPLICATION FIELD: medicine, biology, consumer goods

PUBLICATIONS:

- 1) A. Petica, S. Gavriiliu, M. Lungu, N. Buruntea, C. Panzaru, ***Colloidal silver solutions with antimicrobial properties***, Materials Science and Engineering B, vol. 152, Issues 1-3, Aug. 2008, p. 22 – 27, ISSN 0921-5107, ISI impact factor: 1,33 (in 2007).
- 2) S. Gavriiliu, M. Lungu, E. Enescu, F. Grigore, C. R. Ionescu, ***Stable colloidal silver solutions for different applications***, Optoelectronics and Advanced Materials – Rapid Communications (OAM – RC), vol. 3, issue 6, June 2009, p. 634 – 637, ISSN 1842-6573, ISI impact factor: 0,333 (in 2009).
- 3) C. Gaidau, A. Petica, C. Ciobanu, T. Martinescu, ***Investigation on silver nanoparticles interaction with collagen based materials***, Journal of Optoelectronics and Advanced Materials, Vol. 11, No. 6, (2009) p. 845 - 851, ISSN 1454-4164, ISI impact factor: 0,577 (in 2008).
- 4) C. Gaidau, A. Petica, C. Ciobanu, T. Martinescu, ***Investigation on antimicrobial activity of collagen and keratin based materials doped with silver nanoparticles***, Romanian Biotechnological Letters, Vol. 14, No.5, 2009, p. 4662 - 4665, ISSN 1224-5984.
- 5) A. Petica, S. Gavriiliu, N. Buruntia, ***Ecological method for obtaining of some colloidal silver solutions***, RO Patent Request, OSIM No. A/0034 of 18.04.2008.

CAPITALIZATION: Contract No. 2024/2009 with **SC CRIN IMPEX SRL**.
Contract object: delivery of colloidal silver solutions obtained by chemical method

CHEMICAL PHOTOCATALYTIC AND BIOCIDAL NANOCOMPOSITE POWDERS AND SLIMES



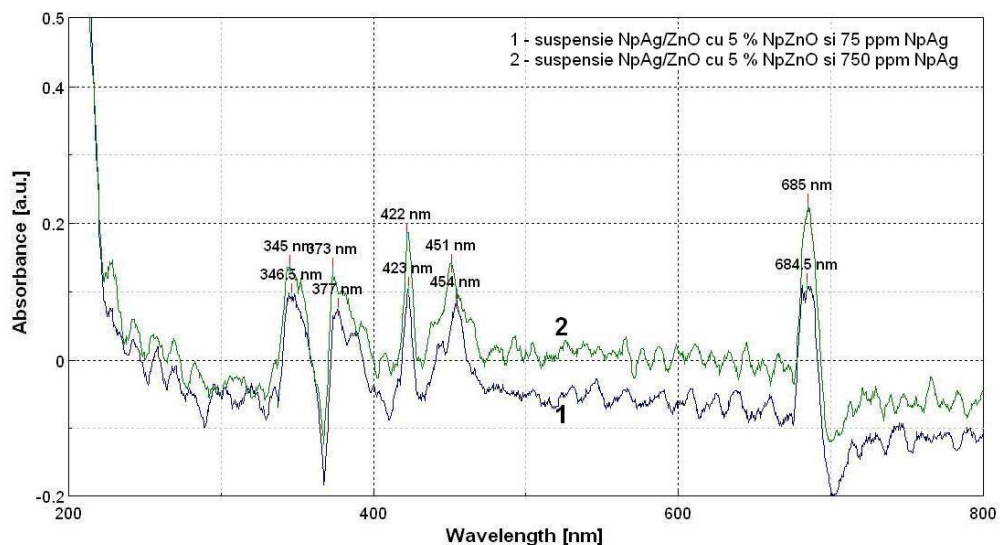
GENERAL DESCRIPTION:

New materials in form of ultrafine powders or slimes of light to brown yellow colour based on zinc oxide nanoparticles (NpZnO) or titania (NpTiO₂) covered with silver nanoparticles (NpAg). The high biocidal and self-cleaning activity is based on the synergism between antimicrobial and photocatalytic properties of the component nanoparticles.

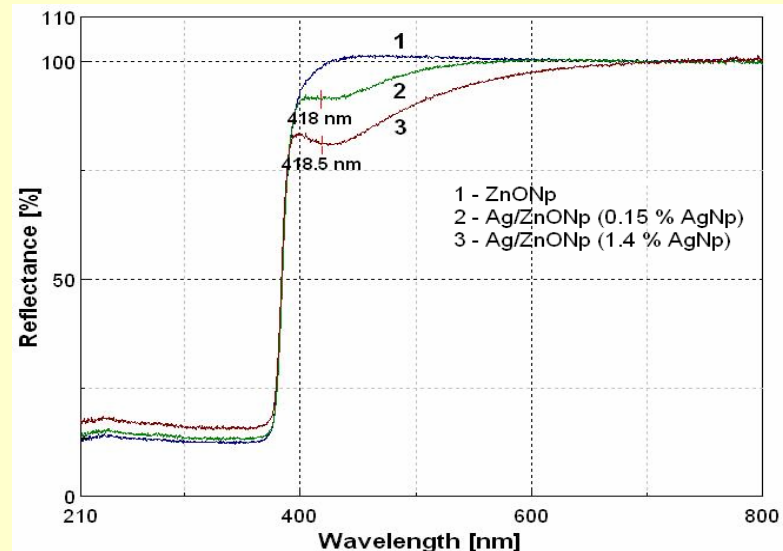


TECHNICAL CHARACTERISTICS:

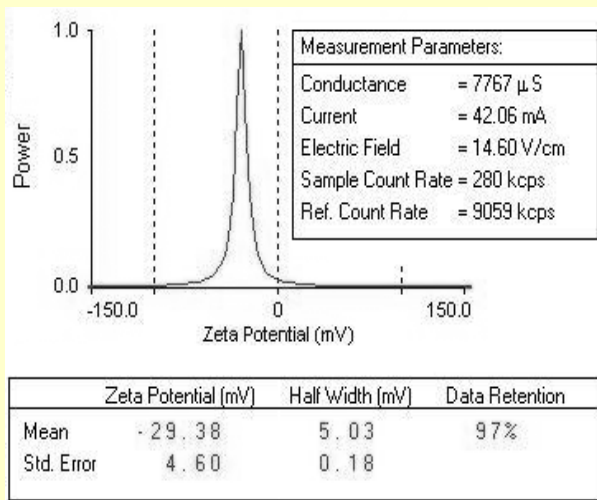
- NpAg/ZnO or NpAg/TiO₂ composition: in various proportions, depending on the application field
- high degree of finesse and dispersion of components
- broad antimicrobial spectra
- photocatalytic and self-cleaning properties
- dispersible in water
- biocompatible and ecological



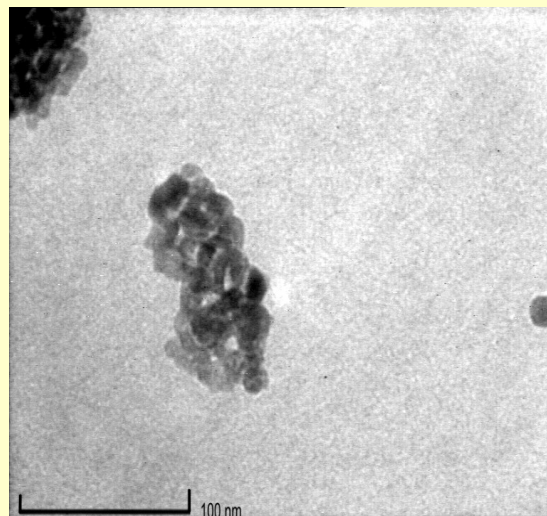
UV-Vis absorption spectra of NpAg/ZnO suspensions



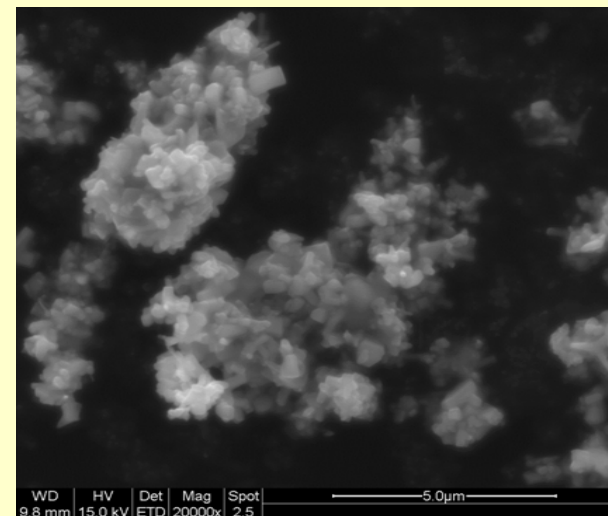
UV-Vis reflectance spectra of ZnONp and Ag/ZnONp composite powders



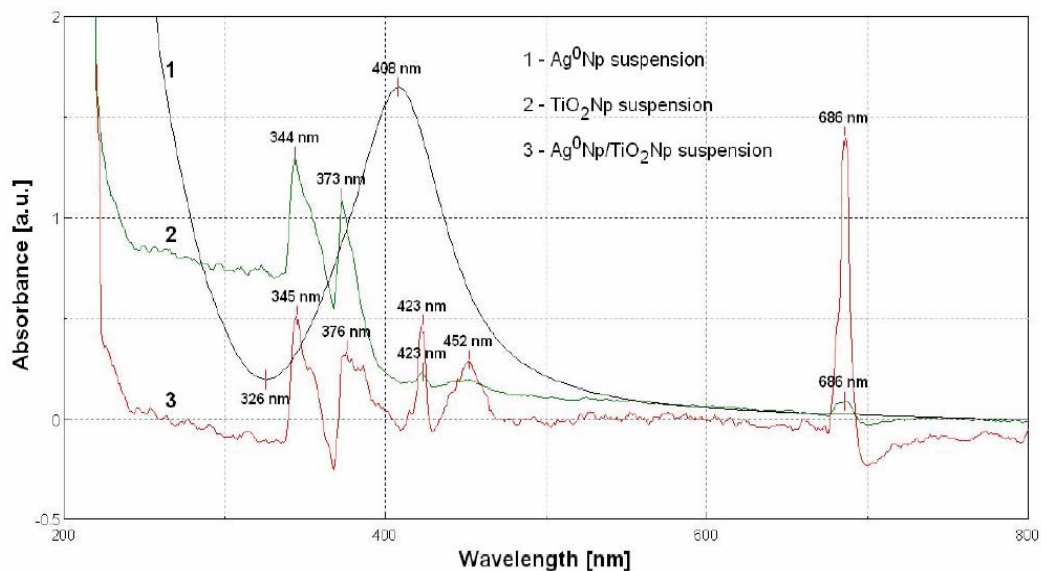
Zeta potential of NpAg/ZnO suspension



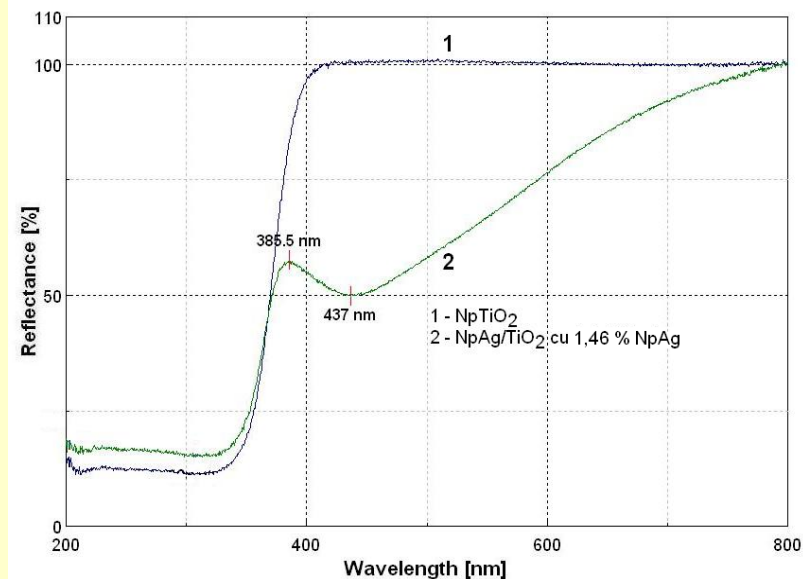
TEM image of Ag/ZnO composite nanopowders



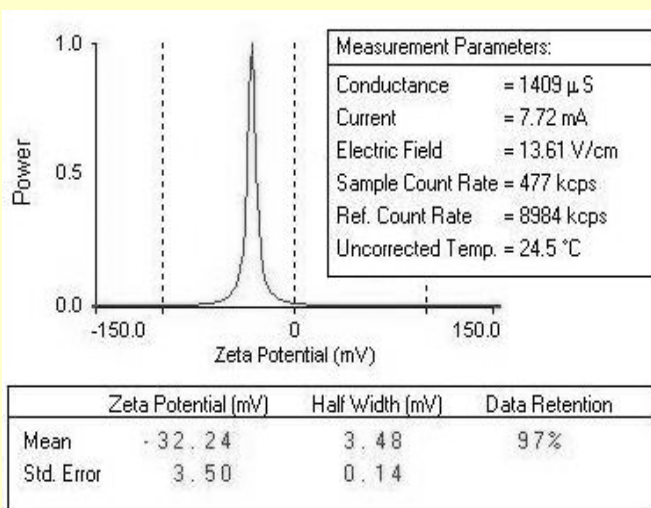
SEM image of Ag/ZnO composite nanopowders



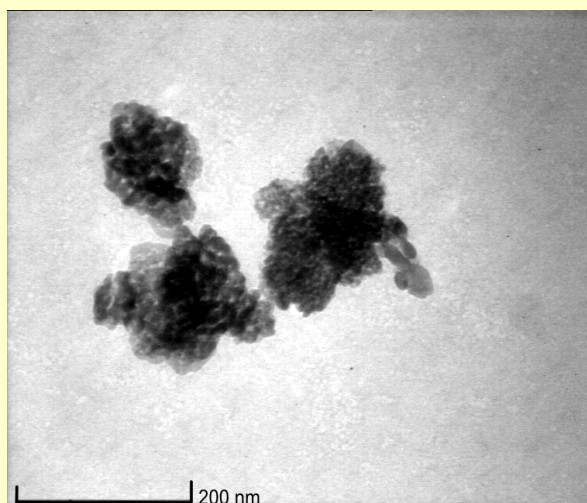
UV-Vis absorbance spectra of AgNp, TiO₂Np and AgNp/TiO₂ suspensions



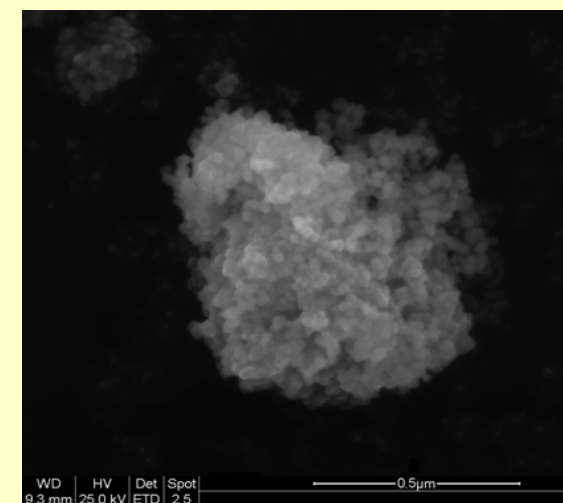
UV-Vis reflectance spectra of TiO₂Np and Ag/TiO₂Np composite powders



Zeta potential of NpAg/ TiO₂ suspension



TEM image of Ag/TiO₂ composite nanopowders



SEM image of Ag/TiO₂ composite nanopowders

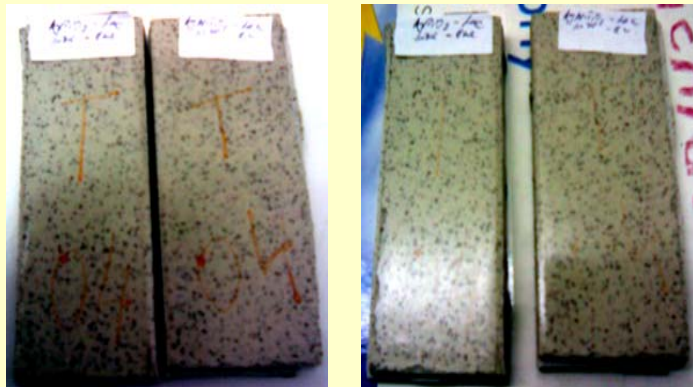
NANOSTRUCTURED DISPERSE SYSTEMS OF Ag-TiO₂/N-TiO₂ TYPE



GENERAL DESCRIPTION:

New materials in form of slimes based on Ag-TiO₂/N-TiO₂Np that combine the photocatalytic property of TiO₂Np and antimicrobial properties of NpAg.

These materials are obtained by an innovative electrochemical method with a minimum impact upon the environment.



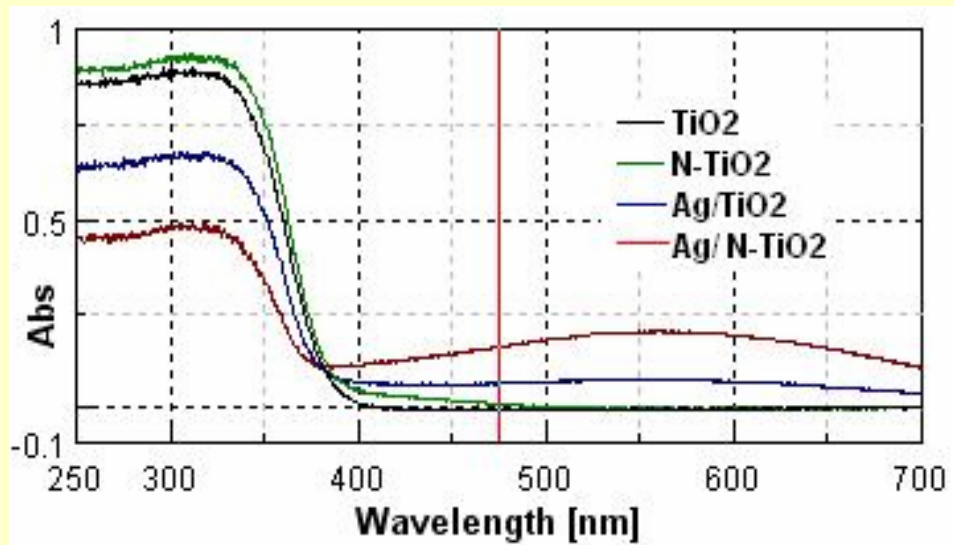
a)

b)

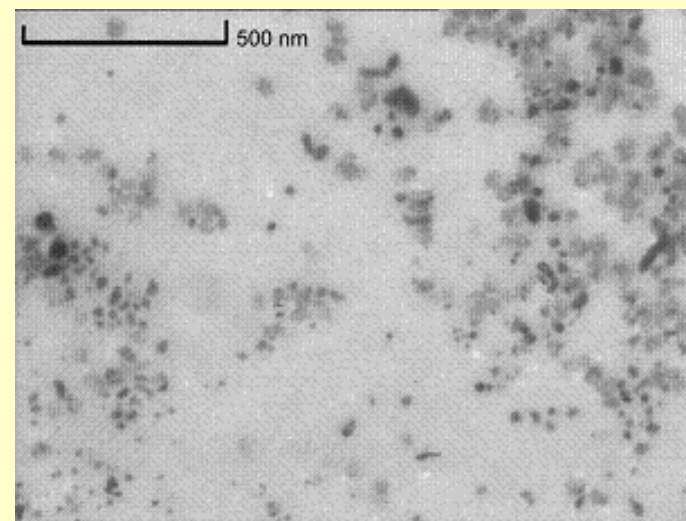
Aspect of ceramic samples covered with an ecological lacquer containing Ag/TiO₂Np, respectively Ag/N-TiO₂Np (a) and after 72 hours of artificial illumination (visible domain) (b)

TECHNICAL CHARACTERISTICS:

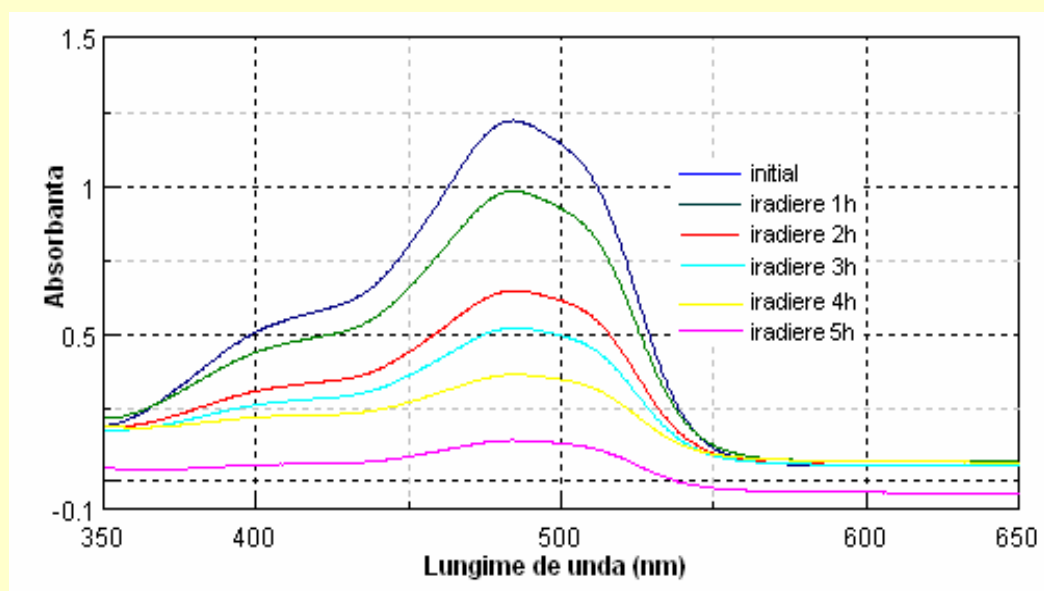
- TiO₂/N-TiO₂ content: 1 – 10%;
- NpAg content: 1 – 8 wt. % against TiO₂ or N-TiO₂;
- average diameter of nanocomposite powders: 20 – 60 nm
- zeta potential: -65...-35 mV;
- broad antimicrobial spectra
- high photocatalytic and self-cleaning activities
- ecological



UV-Vis absorbance spectra of TiO_2 , N-TiO_2 , Ag/TiO_2 and Ag/N-TiO_2 suspensions



TEM image of $\text{Ag-TiO}_2/\text{N-TiO}_2$ composite powder



Photocatalytic degradation of Orange II dye

APPLICATION FIELD: obtaining of antimicrobial products in medicine, cosmetics and consumer goods (dyes, coverings with self-cleaning properties, textiles, leathers, skinfurs, etc.)

PUBLICATIONS:

- 1) S. Gavrilu, M. Lungu, F. Grigore, N. Buruntia, C. Groza, ***New composite powders with high antifungal properties***, Optoelectronics and Advanced Materials – Rapid Communications (OAM – RC), vol. 3, issue 8, August 2009, p. 795 – 799, ISSN 1842-6573, ISI impact factor: 0,333 (in 2009).
- 2) S. Gavrilu, M. Lungu, L. C. Gavrilu, F. Grigore, C. Groza, ***Antimicrobial Colloidal Suspensions of Silver-Titania***, The Open Chemical and Biomedical Methods Journal, 2009, vol. 2, p. 77 – 85, ISSN 1875-0389, indexata in Chemical Abstracts, Google, Google Scholar, Open J-Gate, Genamics Journal Seek.
- 3) L. Anicai, A. Petica, S. Gavrilu, ***Electrochemical method for obtaining of some ecological nanostructured disperse systems with photocatalytic and antimicrobial activity***, RO Patent Request, OSIM No. A/00961 of 23.11.2009.
- 4) S. Gavrilu, M. Lungu, E. Enescu, ***Composite nanostructures of silver- metal oxide type with antimicrobial activity and method for their obtaining***, RO Patent Request, OSIM No. A/01079 of 23.12.2009.

SILVER NANOPARTICLES/BETA-CALCIUM TRIPHOSPHATE

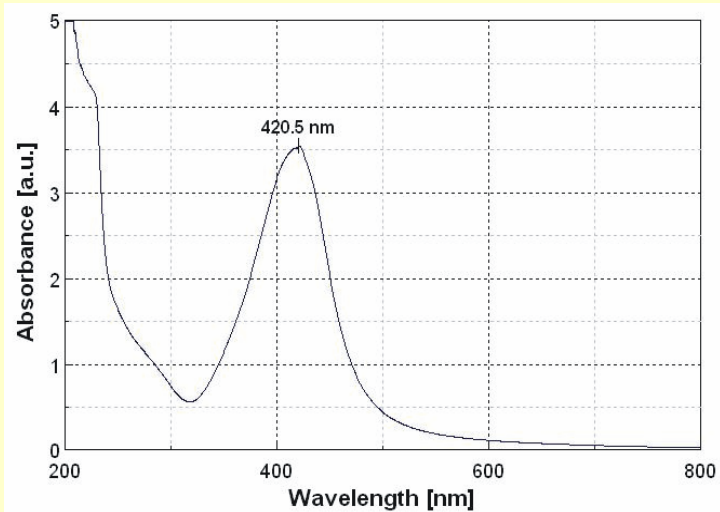


GENERAL DESCRIPTION:

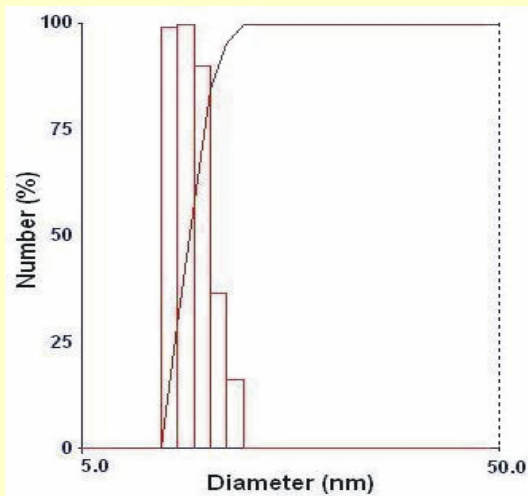
Composite biomaterial in form of a 3D porous ceramic structure doped by dipping with bioactive NpAg that are uniformly and fine dispersed in a β -TCP matrix, conferring them a high antimicrobial and regenerative activity in bone repairs.

TECHNICAL CHARACTERISTICS:

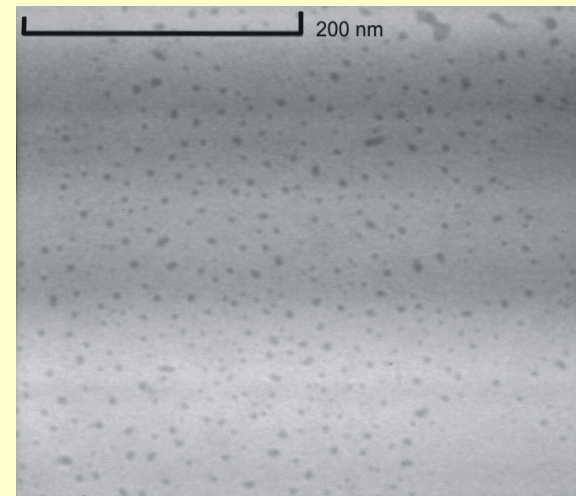
- Ag nanoparticle content: 2.5...60 ppm
- mean diameter of NpAg: 10 nm
- relative density: 2.7 g/cm³
- porosity: 75 %
- pores size: 150 - 500 μ m



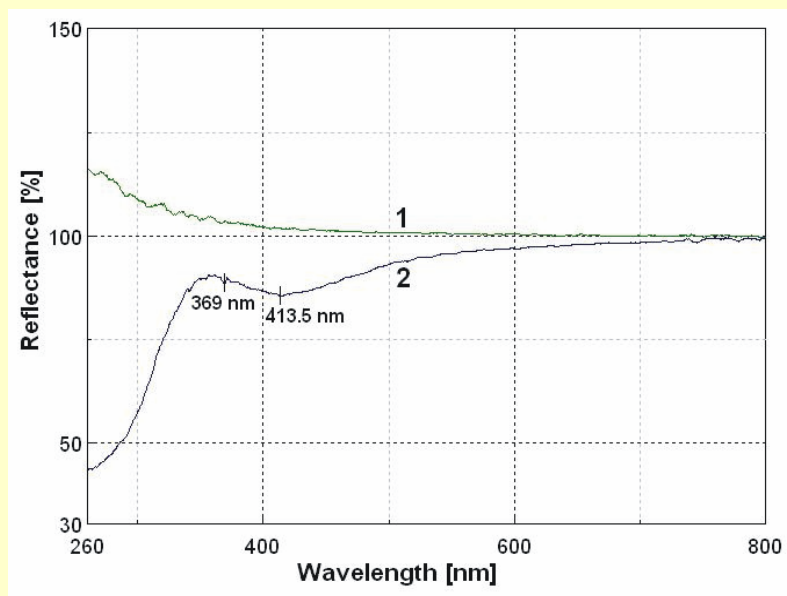
UV-Vis absorbance spectrum of NpAg



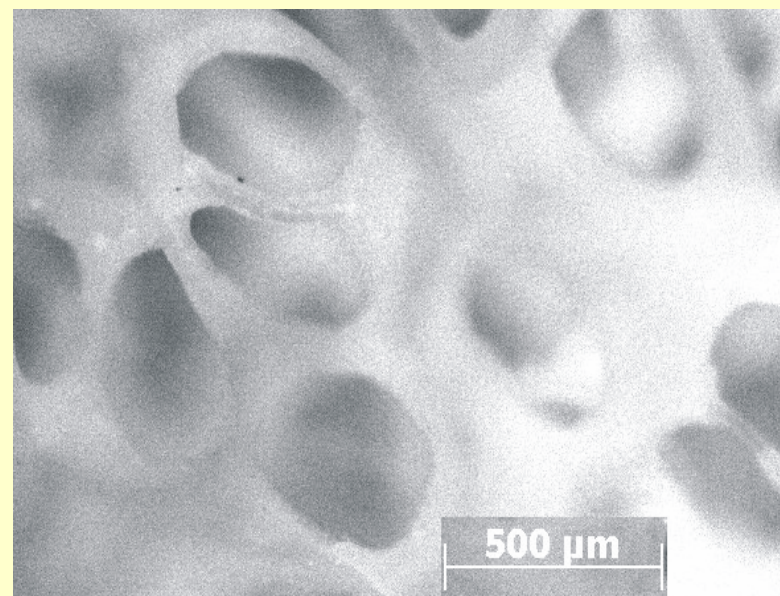
Grain size distribution of NpAg



TEM image of NpAg



Reflectance spectra of undoped (1) and doped (2) biomaterial



Optical image of the final product structure

FUNCTIONAL CHARACTERISTICS:

- ❖ multifunctional nanocomposite biomaterial with a high biological activity:
 - antibacterial activity against a broad spectrum of bacteria, especially *Staphylococcus aureus*, which is involved in osteomyelitis disease;
 - regenerative activity of the bones;
 - biocompatible and bioresorbable;
 - total delivery of the NpAg simultaneously with bioresorption process.
- ❖ simple, ecological, efficient and reproducible process.

APPLICATION FIELD: orthopedic surgery, dentistry surgery, tissue engineering

PUBLICATIONS:

- 1) F. Grigore, E. Andronescu, S. Gavrilu, M. Lungu, Ch. Tardei, ***Characterizations of the β -TCP suspensions***, Revista de Chimie, Vol. 60, Nr. 10, oct. 2009, p. 1107 – 1109, ISSN 0034-7752, ISI impact factor: 0,389 (in 2008).
- 2) F. Grigore, S. Gavrilu, M. Lungu, ***Advanced ceramic nanocomposite for bone repairing and method for its obtaining***, RO Patent Request, OSIM No. A/00847 din 3.11.2008.

SILVER NANOPOWDERS



GENERAL DESCRIPTION:

Fine powders of dark blue colour, obtained by a chemical method of Ag^+ ions reduction in different mediums, in presence of some proper dispersion and stabilization agents.

TECHNICAL CHARACTERISTICS:

- average diameter of NpAg: 10 nm
- narrow ranges of grain size distribution
- particles shape: spherical

APPLICATION FIELD:

- ✓ medicine
- ✓ biology
- ✓ obtaining of antimicrobial products in cosmetics and consumer goods

ACRYL-STIRENE POLYMER DYE DOPED WITH COMPOSITE NANOPOWDERS OF SILVER/TITANIA



GENERAL DESCRIPTION:

New product in form of viscous emulsion of light to brown yellow colour based on acryl-stirene polymers with NpAg/TiO₂ composite powders. The product presents a high bioactivity based on the synergism between antimicrobial and photocatalytic properties of component nanoparticles.

PRODUCT REALIZED IN PARTNERSHIP WITH SC ICAA SA BUCHAREST

TECHNICAL CHARACTERISTICS:

- solid content: min. 45 wt. %
- NpAg/TiO₂ content: 20...30 wt. %
- density: 1.5 g/cm³
- Brookfield viscosity at 25°C: min. 6000 cP
- covering layers: 2
- layer aspect: smooth, opaque
- high degree of finesse and dispersion of components
- broad antimicrobial spectra
- photocatalytic and self-cleaning properties
- dispersible in water
- ecological product

APPLICATION FIELD: obtaining of covering products antibacterial, antifungal and self-cleaning functionalized for interior and exterior finishings of buildings.

PUBLICATION:

R. Gardu, A. Pica, S. Gavrilu, M. Lungu, A. Ciocanete, *Antimicrobial ecological nanocoverings*, RO Patent Request, OSIM No. A/00841 of 11.12.2007.

PRODUCT REALIZED IN PARTNERSHIP WITH SC ICAA SA BUCHAREST

ANTIBACTERIAL AND ANTIFUNGAL PRODUCTS DOPED WITH SILVER NANOPARTICLES



ANTIBACTERIAL AND ANTIFUNGAL PRODUCTS DOPED WITH SILVER NANOPARTICLES

GENERAL DESCRIPTION:

Common value added products obtained by antimicrobial functionalization with bioactive silver nanoparticles (NpAg).
The methods applied at doping are ecological.

TECHNICAL CHARACTERISTICS:

- average diameter of NpAg: 10 nm
- high antimicrobial activity
- oligodynamic bioactivity
- biocompatibility

APPLICATION FIELD:

- ✓ domestic use
- ✓ public places
- ✓ medicine
- ✓ cosmetics

HYDROGEL WITH SILVER NANOPARTICLES



GENERAL DESCRIPTION:

Gel of light to brown yellow colour in form of bioactive silver nanoparticles (NpAg), uniform and fine dispersed in a biocompatible and biodegradable polymeric matrix.

It removes easily with water and not stain.

The synthesis method is ecological and efficient.

TECHNICAL CHARACTERISTICS:

- NpAg content: 10 ppm ... 1 %
- average diameter of NpAg: 10 nm
- biocompatible
- broad antimicrobial spectra
- regenerative action upon skin and hair follicles
- oligodynamic bioactivity.

PRODUCT REALIZED IN PARTNERSHIP WITH ICMM "P. PONI" IASSY



CASE STUDY UPON A DERMATOSIS: before and after curing with NpAg doped hydrogel

APPLICATION FIELD: medicine and cosmetics, at treatment of some skin diseases (burns, dermatoses, wounds).

PUBLICATION:

C. Ciobanu, S. Gavrilu, M. Lungu, L. Gavrilu, L. C. Ciobanu, ***Polyurethane Gel with Silver Nanoparticles for the Treatment of Skin Diseases***, The Open Chemical and Biomedical Methods Journal, 2009, vol. 2, p. 86 – 90, ISSN 1875-0389, indexed in IDB.

PRODUCT REALIZED IN PARTNERSHIP WITH ICMM “P. PONI” IASSY

NANOSTRUCTURED MATERIALS FOR ELECTRICAL ENGINEERING APPLICATIONS

- ❖ Silver nanopowders
- ❖ Silver nanoparticles deposited on microcrystalline silver powders
- ❖ Silver nanoparticles deposited on tin oxide powders

SILVER NANOPOWDERS



GENERAL DESCRIPTION:

Fine powders of dark brown to black colour, depending on the sizes of silver nanoparticles (NpAg) obtained by a chemical reduction method of Ag^+ ions in different mediums in presence of some proper tensioactive agents.

TECHNICAL CHARACTERISTICS:

- average diameter of NpAg: 50...150 nm
- narrow ranges of grain size distribution
- particles shape: spherical

APPLICATION FIELD: obtaining of conductive pastes in electronics

SILVER NANOPARTICLES DEPOSITED ON MICROCRYSTALLINE SILVER POWDERS

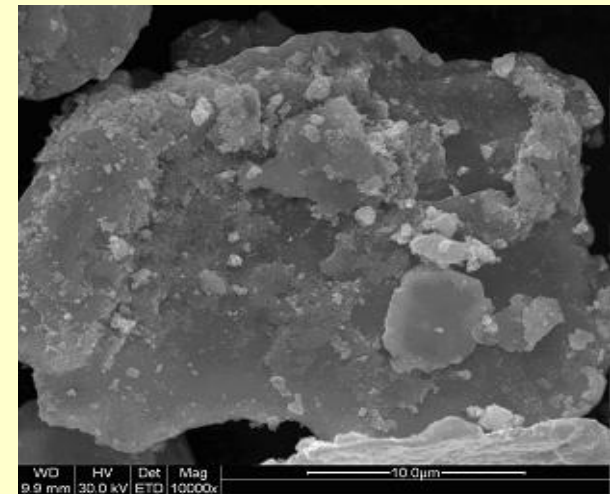


GENERAL DESCRIPTION:

New ecological material in form fine powders of grey to brown colour with improved sintering ability based on silver nanoparticles (NpAg) deposited on silver microparticles (MpAg) by a chemical, ecological and efficient method.

TECHNICAL CHARACTERISTICS:

- average diameter of NpAg: 50 nm
- D_{FSSS} of NpAg/MpAg powder: 1.1 μm
- maximum diameter of NpAg/MpAg powder: 32 μm
- bulk density of NpAg/MpAg powder: 0.98 g/cm^3
- tapped density of NpAg/MpAg powder: 2.7 g/cm^3
- sintering temperature of NpAg/MpAg powder: 200...600 $^{\circ}\text{C}$



APPLICATION FIELD: obtaining of conductive products in electrical engineering and electronics (electrical contacts, pastes etc.)

PUBLICATION:

S. Gavrilu, M. Lungu, F. Grigore, D. Donescu, M. Ghiurea, *Nano/Micro Silver Powders for Electronic Materials*, Journal of Optoelectronics and Advanced Materials, Vol. 10, No. 12, Dec. 2008, p. 3247 – 3250, ISSN 1454-4164, ISI impact factor: 0,577 (in 2008)

SILVER NANOPARTICLES DEPOSITED ON TIN OXIDE POWDERS

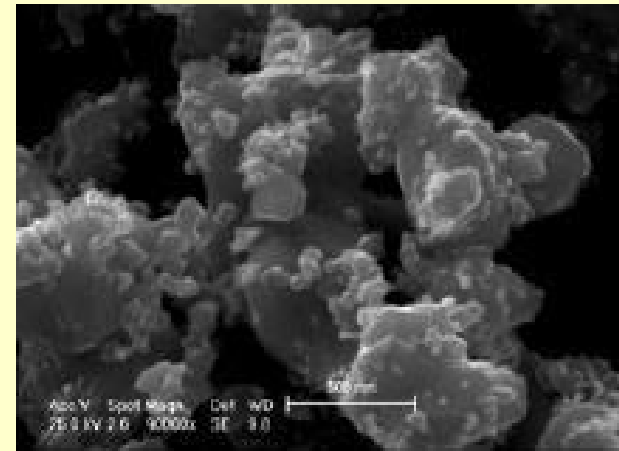


GENERAL DESCRIPTION:

New ecological material in form fine powders of grey to brown colour with improved sintering ability based on silver nanoparticles (NpAg) deposited on tin oxide microparticles (MpSnO_2) by a chemical, ecological and efficient method.

TECHNICAL CHARACTERISTICS:

- NpAg content: 0.5 ...1 %
- average diameter of NpAg: 50 nm
- D_{FSSS} of NpAg/ MpSnO_2 powder: 0.5 μm
- bulk density of NpAg/ MpSnO_2 powder: 1.16 g/cm^3
- high degree of finesse and dispersion
- improved sintering ability



APPLICATION FIELD: obtaining of electrical contacts in electrical engineering

PUBLICATION:

S. Gavrilu, M. Lungu, *Silver-metal oxide powder mixtures for conductive materials and method of their obtaining*, RO Patent Request, OSIM No. A/00098 of 6.02.2008

2008 **ARGA**
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 proizvoda i tehnologija
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 and technologies
 Zagreb, Zagrebački velesajam, 16. 21. rujna 2008.

ZLATNA PLAKETA
 National Institute for Research and Development in Electrical Engineering ICPE-CA
 Bucharest, ROMANIA

Ecological method for silver colloidal solutions obtaining
 Petica Aurora, Gavrilu Stefania, Buruntia Nicoleta
 U Zagrebu, 20. rujna 2008.

Predsjednik ocjenjivačkog suda
 prof. dr. sc. Petar Duhovnik
 Predsjednik Udruge Inovatori Hrvatske
 dr. sc. Stanišavlje Čalavec

HAMAG HET

Medalia de Aur
 se acordă

INCIE ICPE-CA

Procedeu ecologic de obtinere a unor soluții coloidale de argint
 Autori: PETICA AURORA, GAVRILIU ȘTEFANIA, BURUNTIA NICOLETA
 cu ocazia participării la ediția a XII-a

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SALONUL INTERNAȚIONAL DE INVENȚIĂ PRO INVENT ediția a VI-a, 2008, Cluj-Napoca

DIPLOMA
 DE EXCELENȚĂ și medalia de argint

Se acordă Kappel Wilhelm, Mihai Mihaiescu, Stefania Gavrilu, Magdalena Lungu, Mircea Izat, George Zamescu, Eros Patroi, Remus Erdei, Nentiu Jenica, Georgescu Gabriela, Nicolae Verga, Ioana Jitaru, Horia Iovu, Teodora Malauru, Lucian Paslaru-Danescu, Elena Macameto, Sebastian Soltan, Victor Stoica, Ioan Pulfăa

pentru Grup de invenții
 INSTITUTUL NAȚIONAL DE CERCETARE DEZVOLTARE PENTRU INGINERIE ELECTRICA ICPE-CA

PREȘEDINTELE JURIEI CLUJ
 Prof. Ing. Dr. RADU Munteanu
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for the innovation
 Silver - metallic oxides powder mixtures for conductive materials and procedure of their obtaining

Silver medal

THE PRESIDENT OF THE INTERNATIONAL JURY THE PRESIDENT

Medalia de Bronz
 se acordă

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Amestecuri de pulberi din argint-oxizi metalici pentru materiale conductoare și procedeu de obtinere a acestora
 Autori: GAVRILIU ȘTEFANIA MARIA, LUNGU MAGDALENA VALENTINA
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