

COMPUSI SILOXANICI CA PRECURSORI PENTRU NANOMATERIALE

ACASA

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ECHIPA

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BUGET

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* 2007

* 2008

* 2009

* 2010

ICMPP Iasi

CNCSIS

PNCDI II – Program IDEI

Proiect ID_233

Contract nr. 5/28. 09.2007

Director de proiect: Dr. Carmen Racles

Perioada de derulare: 1 oct. 2007 – 30 sept. 2010

Buget total: 750 000 RON

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Plecand de la cunoscuta incompatibilitate a polisiloxanilor cu majoritatea polimerilor organici, proiectul isi propune cresterea gradului de cunoastere privind posibilitatea de control a morfologiei bifazice in sistemele siloxan-organice, pentru a obtine materiale nanostructurate cu proprietati speciale si potentiale aplicatii in domenii de varf. Se vor investiga noi perspective in ce priveste relatiile structura-morfologie-proprietati in materialele multifunctionale continand siloxan. Vor fi obtinute noi sisteme organo-siloxanice (copolimeri, blenduri polimerice si sisteme polimer-disperse) si vor fi studiate pentru a observa nano-organizarea in stare solida, in solutie sau topitura si pentru a intelege influenta morfologiei bifazice asupra proprietatilor globale si de suprafata. Sinteza copolimerilor continand siloxan cu diverse arhitecturi va fi realizata prin reactii de policondensare sau poliaditie, plecand de la macromeri preformati. Structurile vor fi confirmate prin metode spectrale (RMN, FT-IR) si proprietatile lor vor fi investigate prin DSC, DMA, spectroscopie UV-VIZ, microscopie optica in lumina polarizata (POM). Morfologia bifazica si nanostructurarea vor fi evidentiata prin analize TEM, SEM, AFM, XRD si XPS. Vom investiga potentialul unor surfactanti si cristale lichide siloxanice pentru utilizare ca nanoreactoare sau nanocontainere. Se va testa prepararea unor nano-obiecte, inclusiv nanoparticule polimerice prin nanoprecipitare sau reactii de polimerizare ori policondensare in nanoreactoare constand din structuri supramoleculare bifazice siloxan-organice. Nano-materialele rezultate vor putea fi folosite fie ca faza dispersa in blenduri polimerice si sisteme polimer-disperse, fie ca vehicule pentru substante active. Se va urmari aplicarea cu precadere a materialelor biocompatibile sau biodegradabile si folosirea formularilor in faza apoasa, avand drept tinta finala aplicatii in domeniul medical si al produselor de ingrijire.

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Dr. Carmen Racleş - director de proiect

Dr. Maria Cazacu

Dr. Vasile Cozan

Dr. Aurelia Ioanid

Tineri doctoranzi:

Mihaela Alexandru

George Stiubianu

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Obiectiv general: *cresterea gradului de cunoastere in domeniul controlului morfologiei bifazice in sistemele continand siloxani, cu scopul de a obtine materiale nanostructurate.*

Obiectivele specifice:

- sinteza de noi sisteme bi/multifazice continand siloxani (copolimeri, amestecuri de polimeri, sisteme polimer-disperse) si studiul lor din punct de vedere morfologic, pentru intelegerea influentei morfologiei bifazice asupra proprietatilor globale si de suprafata;
- testarea unor abordari neconventionale pentru sinteza de nanoparticule, in special sinteza in nanoreactoare pe baza de compusi siloxan-organici;
- folosirea nanoparticulelor polimerice in scopuri neconventionale (de exemplu ca faza dispersa in amestecuri);
- explorarea formularilor in faza apoasa continand nanoreactoare si nanocontainere pe baza de siloxani pentru aplicatii tinta, precum sisteme de transport si eliberare de medicamente, acoperiri, vopsele, produse de ingrijire personala.

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BUGETUL PROIECTULUI (RON)

2007	2008	2009	2010	TOTAL
65 000	250 000	250 000	185 000	750 000

REPARTIZAREA CHELTUIELILOR – 2007

Denumire capitol buget	Cheltuieli de personal	Cheltuieli indirecte	Mobilități	Cheltuieli de logistică	Total
Valoare (RON)	29000	18571	0	17429	65000

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2007

Obiective propuse

Sinteza de compusi siloxanici multifunctionali

Caracterizarea fizico-chimica a compusilor siloxan-organici

Rezultate semnificative

- Noi precursori organici mezogeni;
- Copolimeri siloxan-organici cu grupe chelatoare;
- Copolimeri siloxanici cu grupe functionale pendante;
 - obtinuti prin reactii de condensare pe catena;
 - obtinuti prin reactii de aditie (hidrosililare) pe catena;
- Compusi multifunctionali nanoporosi.
- Caracterizarea structurala prin metode spectrale
- Caracterizarea termica /termotropa.

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REPARTIZAREA CHELTUIELILOR – 2008

Denumire capitol buget	Cheltuieli de personal	Cheltuieli indirecte	Mobilități	Cheltuieli de logistică	Total
Valoare (RON)	120000	71428	10000	48572	250000

Echipamente achizitionate:

- Baie de termostatare pentru vascozitate;
- Pompe de vid

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Obiective propuse

1. Studiul morfologiei unor polimeri bifazici continand unitati siloxanice
2. Sinteza de compusi siloxanici amfifili
3. Caracterizarea surfactantilor siloxanici

Rezultate semnificative

- Observatii de microscopie electronica si AFM si corelatii structura-morfologie in cazul unor copolimeri segmentati si unor co-retele amfifile siloxan-organice ;
- Obtinerea si caracterizarea unor noi compusi amfifili siloxan-organici cu potential de surfactanti ne-ionici, avand diverse arhitecturi (macromeri liniari sau ciclici, copolimeri cu grupe functionale pendante), si unitati hidrofile derivate de monozaharide si THMAM ;
- Investigarea unor compusi siloxanici amfifili solubili in apa privind proprietatile de suprafata si capacitatea de stabilizare a nanoparticulelor (teste preliminare prin nanoprecipitare si reactii chimice).

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Obiective 2009

1. Obținerea de nanoparticule polimere prin metode fizice
2. Sinteza unor polimeri în nanoreactoare siloxanice
3. Caracterizarea dimensională a nanoparticulelor obținute

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LUCRARI APARUTE / ACCEPTATE

Reviste cotate ISI

Propuse (2008+2009): 4 / Realizate (iunie 2009): 6; Factor de impact mediu (2007): 1,47

1. C. Racles, M. Cazacu, A. Ioanid, A. Vlad, Micellization of a siloxane-based segmented copolymer and its use as a tool for metal complex nanoparticles, **Macromol. Rapid Commun.** 2008, 29, 1527–1531 (FI = 3,383)
2. M. Cazacu, A. Vlad, A. Airinei, M. Alexandru: Silica encapsulating lanthanum complexes using the sol-gel technique, **Polym. Int.** 57: 1067-1074, 2008 (FI= 1,557).
3. A. Vlad, M. Cazacu, G. Munteanu, A. Airinei, P. Budrugaac: Polyazomethines derived from polynuclear dihydroxyquinones and siloxane diamines, **Eur. Polym. J.** 44, 2668-2677, 2008 (FI = 2,248).
4. C. Racles, M. Alexandru, M. Cazacu, A. Ioanid, T. Hamaide: Obtention des elastomeres silicones en nanoreacteurs siloxane-organiques; **Rev. Roum. Chim.** –acceptata (FI=.0,208)
5. E. Avram, V. Cozan: Modified polysulfones with N-phenacyl and N-acetoxyalkyl-4,4'-bipyridinium pendant groups, **Materiale Plastice** 45 (3), 2008, 241-245 (FI = 0,404).
6. Carmen Racles, Maria Cazacu, Gabriela Hitruc, Thierry Hamaide: On the feasibility of chemical reactions in the presence of siloxane-based surfactants; **Colloid Polym Sci** (2009) 287:461–470 (FI =1,62).
7. Vasile Cozan, Mihaela Avadanei, Elena Perju, Daniel Timpu: FTIR investigations of phase transitions in an asymmetric azomethine liquid crystal; **Phase Transitions** (acceptata) (FI = 0,865).

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ACASA**REZUMAT****ECHIPA****OBIECTIVE****BUGET****REZULTATE*** **2007*** **2008*** **2009*** **2010****Reviste indexate in alte baze de date internationale****Propuse (2008+2009): 4 / Realizate (iunie 2009): 4**

1. C. Racles: Siloxane-Containing Liquid Crystalline Polymers, in Advances in Functional Heterochain Polymers, (M. Cazacu –Ed.), Nova Science Publishers, 2008. ISBN: 978-1-60456-599-7.
2. Carmen Racles: Polysiloxanes and siloxane-organic copolymers for nanotechnologies; in Functional Polymeric Materials Designed for Hi-Tech Applications, (M.Nechifor ,Ed.) Ed. Research Signpost, Kerala, India, acceptata 2010: ISBN: 978-81-7895-448-6.
3. V. Cozan, M. ciobanu, L. Marin: Aromatic copoly(ethersulfone)s, in Functional Polymeric Materials Designed for Hi-Tech Application (M.Nechifor ,Ed.), Ed. Research Signpost, Kerala, India, acceptata 2010: ISBN: 978-81-7895-448-6.
4. Carmen Racles, Thierry Hamaide, Etienne Fleury : Siloxane-containing compounds as polymer stabilizers; in Polymer Research Developments: Amphiphillic Block Copolymers, Polymer Aging, Block Copolymers/Polymer Stabilizers, (F. Columbus – Ed.), Nova Science Publishers

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Comunicari la manifestari stiintifice:

1. C. Racles, M. Cazacu, A. Ioanid, A. Vlad: Synthesis of polymer nanoparticles in siloxane-containing nanoreactors, Comunicare *6th International Symposium Molecular Order and Mobility in Polymer Systems –St. Petersburg, iunie 2008*
2. C. Racles, M. Cazacu, A. Ioanid: Siloxane-containing surfactants and their use for stabilization of nanoparticles, Poster- *22nd General Conference of the Condensed Matter Division of the EPS (CMD22) Roma, august 2008*
3. Mihaela Alexandru, Carmen Racles, Maria Cazacu: Siloxane-organic hybrids. Evaluation of some surface properties by tensiometry Comunicare - *International Conference on Materials Science and Engineering, February 26-28, 2009, Brasov, Romania*
4. George Stiubianu, Grigoras Cristian, Carmen Racles, Maria Cazacu: New materials developed on the cellulose and siloxane derivatives. Preparation and properties evaluation Comunicare - *International Conference on Materials Science and Engineering, February 26-28, 2009, Brasov, Romania*

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Petru Poni Institute of
Macromolecular Chemistry,
Iasi

National
University Research
Council

PNC DI II – *IDEAS* Program

Project ID_233

Contract no. 5/28. 09.2007

Running period: 1 oct. 2007 – 30 sept. 2010

Total budget: 750 000 RON

Principal investigator: Dr. Carmen Racles

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Starting from the incompatibility of polysiloxanes with most of the organic polymers, the project aims to increase the knowledge on tailoring the biphasic morphology in siloxane-containing systems, in order to obtain nanostructured materials with special properties and potential top-field applications. We will try to bring new insights into the structure-morphology - properties relationships of siloxane-containing multifunctional materials. New organo-siloxane systems (copolymers, polymer blends, polymer-dispersed systems) will be obtained and studied to observe nano-organization in solid state, in melt or in solution and to understand the influence of the biphasic morphology over bulk and surface properties. The synthesis of siloxane-containing copolymers with various architectures will be realized by polycondensation or polyaddition reactions, starting from preformed macromers. The structures will be confirmed by spectral methods (NMR, FT-IR) and their properties will be investigated by DSC, DMA, UV-VIS spectroscopy, polarized light POM. The biphasic morphology and nanostructuration will be evidenced by TEM, SEM, AFM, XRD and XPS. The potential of siloxane based surfactants or liquid crystals as nanoreactors or nanocontainers will be investigated. We will test the preparation of nano-objects, including polymeric nanoparticles, by nanoprecipitation or polymerization / polycondensation reactions in nanoreactors consisting of biphasic organo-siloxane supramolecular assemblies. The results will be evaluated for subsequent use as disperse phase in polymer blends and as delivery vehicles. Mostly biocompatible / biodegradable materials will be designed and water-based formulations for medical and personal care use will be explored.

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Dr. Carmen Racleş - principal investigator

Dr. Maria Cazacu

Dr. Vasile Cozan

Dr. Aurelia Ioanid

PhD students:

Mihaela Alexandru

George Stiubianu

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General objective: *the increase of knowledge on tailoring the biphasic morphology in siloxane-containing systems, in order to obtain nanostructured materials.*

Specific objectives:

- to synthesize new bi/multiphase siloxane-containing systems (copolymers, polymer blends, polymer- dispersed systems) and to study them from the morphological standpoint, in order to understand the influence of biphasic morphology over bulk and surface properties;
- to test unconventional approaches such as siloxane-based nanoreactors for the synthesis of nano-objects;
- to use polymeric nanoparticles (nano-objects) for unconventional purposes (for example as disperse phase);
- to explore water-based formulations containing siloxane-based nanoreactors and nano-containers for targeted applications, such as drug delivery systems, coatings, paints, personal care.

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PROJECT BUDGET (RON)

2007	2008	2009	2010	TOTAL
65 000	250 000	250 000	185 000	750 000

Budget For 2007

Budget Chapter	Personnel Costs	Indirect Costs	Mobilities	Logistics Costs	Total
Value (RON)	29000	18571	0	17429	65000

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2007

Proposed objectives

1. Synthesis of multifunctional siloxane compounds
2. Characterization of the organo-siloxane compounds

Significant results

- New organic mesogenic presursors;
- Organo-siloxane copolymers with chelate groups;
- Siloxane copolymers with functional pendant groups;
 - obtained by condensation reactions on the backbone;
 - obtained by addition reactions (hydrosilylation);
- Multifunctional nanoporous compounds.
- Structural characterization by spectral methods;
- Thermal / thermotropic characterization.

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Budget For 2008

Budget Chapter	Personnel Costs	Indirect Costs	Mobilities	Logistics Costs	Total
Value (RON)	120000	71428	10000	48572	250000

Equipments:

- Thermostated bath for viscosity measurements;
- Vacuum pumps

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2008

Proposed objectives

1. Study of the morphology of biphasic polymers containing siloxane units;
2. Synthesis of amphiphilic siloxane compounds
3. Characterization of siloxane surfactants

Significant results

- Electron microscopy and AFM observations for siloxan-organic segmented copolymers and amphiphilic co-networks;
- Synthesis and characterization of new siloxan-organic amphiphilic compounds –potentially non-ionic surfactants – having various architectures (linear or cyclic macromers, copolymers with pendant functional groups) and hydrophilic units derived from monosaccharides or THMAM;
- Investigation of water soluble amphiphilic compounds regarding their surface properties and their potential for nanoparticles stabilization (preliminary tests by nanoprecipitation and chemical reactions).

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Proposed objectives

1. Preparation of polymeric nanoparticles by physical methods
2. Polymer syntheses in siloxane nanoreactors
3. Dimensional characterization of the obtained nanoparticles

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Published or accepted articles -ISI

1. C. Racles, M. Cazacu, A. Ioanid, A. Vlad, Micellization of a siloxane-based segmented copolymer and its use as a tool for metal complex nanoparticles, **Macromol. Rapid Commun.** 2008, 29, 1527–1531
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Communications

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