# **ACTIVITY REPORT**

# NEW NANOCOMPOSITES BASED ON BIOCOMPATIBLE POLYMERS AND GRAPHENE FOR DENTAL APPLICATIONS - <u>BIOGRAF (230/2014)</u>

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Work package 3 (2016)

Technical documentation. Patent elaboration. Biological studies.

# <u>A 3.1</u> Preparation of graphene-based nanofillers and their morphological and structural characterization (CO-INCDTIM)

Due to its low cytotoxic effect, graphene-oxide (OG) was selected for addition in dental materials. After preparation, it was combined with oxidic materials (SiO<sub>2</sub> and ZrO<sub>2</sub>) and the corresponding composite materials (OG-SiO<sub>2</sub> and OG-ZrO<sub>2</sub>) were morphologically (TEM) and structurally (XRD; FTIR; UV-Vis) characterized. After that, they were used as nanofillers (in low concentrations) in dental materials.

## <u>A 3.2</u> Optimization of graphene-oxide synthesis (CO-INCDTIM)

The final parameters and the reaction conditions for graphene-oxide synthesis were established. The optimized method was developed. The method ensures the synthesis of graphene oxide having the desired morphological and structural characteristics: thin and transparent sheets (11-12 layers); interlayer distance  $\sim 0.8$  nm.

#### <u>A 3.3</u> Optimization of dental materials synthesis (P1-UBB)

Dental materials with composite materials OG-SiO<sub>2</sub> or OG-ZrO<sub>2</sub> were synthesized and the optimized conditions were establised :

- $\checkmark$  The optimum ratio between the organic and anorganic fillers was 20/80;
- ✓ In order to fulfill the esthetic requirements, low quantities of OG-SiO<sub>2</sub> or OG-ZrO<sub>2</sub> were used (0.2 0.3 wt%)
- $\checkmark$  The optimum time for mixing the organic with the anorganic fillers was 8 hours;
- $\checkmark$  The optimum photopolimerization time was 40 s

## <u>A 3.4</u> Patent elaboration (P1-UBB; CO-INCDTIM)

 A patent request was submitted to OSIM : 'Composite material containing graphene oxide and used for dental restoration' by Marioara Moldovan, Stela Pruneanu, Crina Socaci, Marcela Rosu, Codruta Sarosi, Stanca Cuc, Doina Prodan (OSIM No. A/00902/24.11.2016)

<u>A 3.5</u> Finalizing the biological studies (in vitro): experimental model for testing the adhesion of new restorative materials to the dental hard tissues; electron microscopy and optical microscopy on extracted human teeth (P2-UMF)

The study aimed to evaluate the marginal infiltration on extracted teeth. 15 extracted teeth were included in the study. The teeth were divided into 3 groups of five teeth: *Group 1* - for cavity filling, a material based on graphene oxide and  $ZrO_2$  (GZ2) was used; *Group 2* - for cavity filling, a material based on graphene oxide and SiO<sub>2</sub> (GS4) was used; *Group 3* – the teeth were filled with a light-curing composite trade as Amelogen Plus (Ultradent Products, Inc. USA). The results indicated a

*score* 0 - lack of marginal infiltration, when restoration was performed with GZ2 composite and the commercial Amelogen Plus (*Group 1 and Group 2*). In the case of restoration with the GS4 composite, a marginal infiltration was observed (*score 1- Group 3*).

# <u>A 3.6</u> The feasibility study for industrial research - Part I (P3-Apel Laser SRL)

The feasibility study was elaborated by APEL LASER SRL. The study aimed to provide the appropriateness of introducing into production of a composite material based on biocompatible polymers and graphene, for dental applications.

<u>Act. 3.7</u> Disemination of the research results to PhD students and PostDoc (CO-INCDTIM; P1-UBB; P2- UMF) PhD students and PostDoc were actively involved in most of the research activities.

#### **ISI Papers:**

- Codruta Sarosi, Alexandru Radu Biris, Aurora Antoniac, Stanca Boboia, Camelia Alb, Iulian Antoniac and Marioara Moldovan, *"The nanofiller effect on properties of experimental graphene dental nanocomposites"*, Journal of Adhesion Science and Technology, 2016, VOL. 30, NO. 16, 1779–1794 (IF 0.863) (P1, P2, CO)
- A. MUNTEAN, A. MESAROS, D. FESTILA, M. MOLDOVAN, M. MESAROS, "In Vitro Microleakage Evaluation Around Three Types of Dental Sealants", MATERIALE PLASTICE, Vol.53, No.1, 2016, 166-169 (IF 0.903) (P1)
- M-C Rosu, C Socaci, V Floare-Avram, G Borodi, F Pogacean, M Coros, L Magerusan and S Pruneanu, *Photocatalytic performance of graphene/TiO<sub>2</sub>-Ag composites on amaranth dye degradation*, Materials Chemistry and Physics 179 (2016) 232-241 (IF 2.102) (CO)
- 4. C. Socaci, F.Pogacean, A.R.Biris, M.Coros, M.C.Rosu, L.Magerusan, G.Katona, S. Pruneanu, Graphene oxide vs. Reduced graphene oxide as carbon support in porphyrin peroxidise biomimetic nanomaterials, Talanta, 148(2016)511–517 (IF 4.035) (CO)

#### **Book-chapter**

 'Efectul diferitelor bauturi asupra stabilitatii culorii nanocompozitelor experimentale cu grafene' Codruta Sarosi, Marcela-Corina Rosu, Cristina Prejmerean, Laura Silaghi-Dumitrescu, Stanca Cuc, Doina Prodan, Marioara Moldovan, pg. 95-101, in 'Cercetarea in Medicina dentara- proprietati optice ale dintilor si materialelor dentare', Colorama- Cluj-Napoca; ISBN 606877815-0 (P1; CO)

## Patent Patent

 'Composite material containing graphene oxide and used for dental restoration' M. Moldovan, S. Pruneanu, C. Socaci, M. C. Rosu, C. Sarosi, S. Cuc, D. Prodan (P1, CO)- submitted to OSIM (A/00902/24.11.2016)

# **Conferences**

- CNB 2016, National Conference of Biophysics, 2-4 iunie, 2016, Cluj-Napoca, Romania -Graphene oxide-based composites as biocompatible substrates for cell adhesion and proliferation, M.C. Rosu, C. Socaci, L. Magerusan, F. Pogacean, M. Coros, E. Pall, S. Pruneanu (CO)
- 2. CNB 2016, National Conference of Biophysics, 2-4 iunie, 2016, Cluj-Napoca, Romania *Peroxidase biomimetic materials based on porphyrin and graphene/graphene oxide*, S. Pruneanu, C. Socaci, F. Pogacean, M. Coros, L. Magerusan, M.C. Rosu (CO)
- 3. International Symposium of Dentistry Napoca Biodent 2016, 9-10th May 2016, Cluj-Napoca, Romania, *The effect of different drinks on the color stability of graphene experimental nanocomposites*, C. Sarosi, M. Rosu, C. Prejmerean, L. Silaghi-Dumitrescu, S. Cuc, D. Prodan, M. Moldovan- *Prezentare orala* (P1; CO)
- 4. SCAD 2016: 8<sup>th</sup> Annual Conference of Society for Color and Appearance in Dentistry Chicago, IL, 15-17 Septembrie 2016, *Color change of some experimental nanocomposites after accelerated aging test*, C. Sarosi, M. Rosu, C. Prejmerean, L. Silaghi-Dumitrescu, M. Moldovan (P1; CO)

# **General Conclusions**

#### All the activities foreseen within this work-package were accomplished:

- D7- Material/Patent
- D8- Technical data
- D9- Biological studies
- 4 ISI papers were published
- 1 book-chapter was published
- 1 Patent was submitted to OSIM (nov. 2016)