

PART 2: PROGRAMME PROJECT INFORMATION

2.1. Project No. 5

Title	<i>Material mechanical micro - nano- scaled features and their impact on human safety</i>	
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2.2. Tasks and deliverables

(Describe the project goals and objectives so that the achievements reported below could be placed in context and evaluated)

Project goal: To research early destruction of surface of polymer composite materials, to develop methods of early diagnostics and analyze application of the methods in enterprises.

Task 1 of the 1st Period: *Development of research methods for diagnostics of early destruction of surface of polymer composite materials: the method to research influence of aquatic microorganisms on early destruction of materials* (the task ends in the IV quarter of 2015).

The following was planned in order to accomplish the Task: (1) to develop sample preparation methods and fabricate the samples; (2) to find optimal parameters for optical stimulation of photoelectron emission in order to achieve the maximum signal to noise ratio depending on a type of studied material; (3) to adjust a method for detection of leaching of organic substances from walls of polymeric pipes.

Task 2 of the 1st Period: *Development of research methods for diagnostics of early destruction of surface of polymer composite materials: the method to research visual recognition of early destruction using destruction-induced staining* (the task ends in the IV quarter of 2015).

The following was planned in order to accomplish the Task: (1) to develop sample fabrication methods and fabricate the samples; (2) to develop a method of applying dye containing microcapsules onto the surface of the samples.

Time frame for the tasks is given in Annex 5-A.

The goal and tasks of the 1st Period of the Project were fully achieved. Methods of preparation of fiberglass polymer composites (FPC), polymer composites filled with carbon nanotubes and samples of polymeric pipes were developed. Automatic sheet cutting CNC technique was adjusted for preparation of FPC samples. The developed method of preparation of polymer composites filled with carbon nanotubes consists of component mixing, shaping and drying stages. A special technological device was developed and manufactured for the shaping stage. In order to prepare samples of polymeric pipes, mechanical cutting method was developed. For

each type of the material a specific shape was optimized in order to perform mechanical loading experiments and electron emission measurements. A method of applying dye containing microcapsules onto the surface of composite materials was developed. Spectroscopic method for detection of leaching of organic substances from walls of polymeric pipes was adjusted.

For each type of the material increments of photoemission current during the mechanical loading were studied depending on wavelengths used for photostimulation. A range of wavelengths was chosen which ensures the greatest increment of the current.

The following Deliverables are expected upon the completion of the tasks in the IV quarter of 2015:

Nr.	Task	Deliverable	Responsible partner	Status
1.	Development of research methods for diagnostics of early destruction of surface of polymer composite materials: the method to research influence of aquatic microorganisms on early destruction of materials	Research method for development of diagnostics of early destruction of surface of polymer composite materials using <i>in situ</i> electron emission spectroscopy (31.12.2015)	J. Dehtjars, Institute of Biomedical Engineering and Nanotechnologies, RTU	In progress
2.	Development of research methods for diagnostics of early destruction of surface of polymer composite materials: the method to research visual recognition of early destruction using destruction-induced staining	Research method for development of diagnostics of early destruction of surface of polymer composite materials using destruction-induced staining (31.12.2015)	A. Aņiskevičs, The Institute of Polymer Mechanics of the University of Latvia	In progress

2.3. Description of gained scientific results

(Describe scientific results achieved during reporting period, give their scientific importance)

Tasks of the Project	The main results
1. Development of research methods for diagnostics of early destruction of surface of polymer composite materials: the method to research influence of aquatic microorganisms on early destruction of materials.	Concept of the method developed

The following results were achieved during the report period:

1. A method for sample fabrication was developed and the samples were prepared for loading experiments:
 - a. epoxy resin samples with different concentrations of carbon nanotubes (0%; 0,2%; 0,5%, 1,0%);
 - b. samples of polymeric pipes (rough cutting using bandsaw, fine cutting using vertical milling machine);
 - c. samples of fiberglass polymer composite (rough cutting using bandsaw, fine cutting using vertical milling machine).
2. Equipment for measurement of electron emission was adjusted for measurements in a single electron counting mode during mechanical loading of the samples. Optical stimulation modes have been optimized for measurements of photoelectron emission (PE) from the samples.
3. Initial experiments were performed that demonstrated possibility to detect PE during quasielastic deformation. The experiments have provided evidence that the concept of the method of early diagnostics of destruction can be employed when observing destruction of the surface during quasielastic deformation (Fig.1).

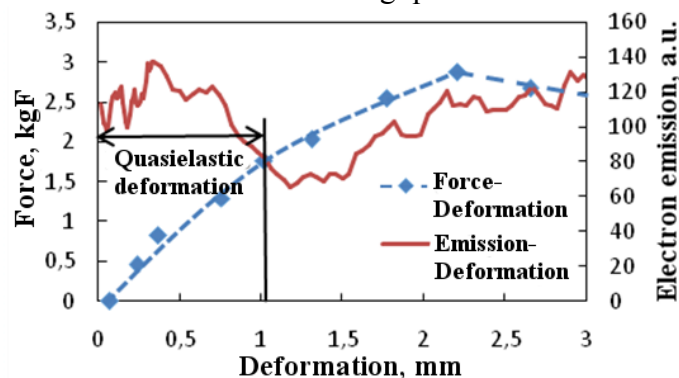


Fig. 1. Changes of photoelectron emission from a polymeric pipe PE-80 during quasielastic deformation

4. Possibility to determine leaching of organic substances (TOC – total organic carbon) which serve as nutrients for microorganisms from Evoqua HDPE (high-density polyethylene) pipes into water was tested. The experiments were performed with different water samples (tap water, tap water with added Cl_2 (0.5 mg/L), ultra-clean deionised water). The results showed that during the observed period the pipes did not leach enough organic substances that could be used as the nutrients by bacteria.

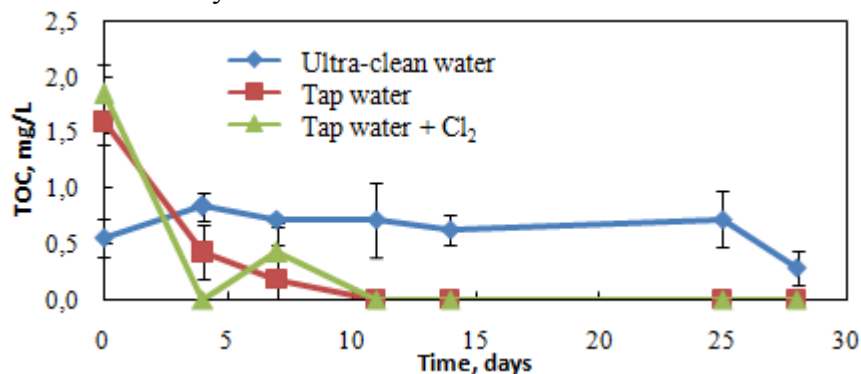


Fig. 2. Changes in TOC concentration using different water samples

5. The experiments have been launched to determine the influence of concentration of the leached TOC on multiplication of natural (*Pseudomonas Fluorescens*) and

faecal (*Escherichia Coli*) microorganisms found in water.

2. Development of research methods for diagnostics of early destruction of surface of polymer composite materials: the method to research visual recognition of early destruction using destruction-induced staining.

Concept of the method developed

The following results were achieved during the report period:

1. A method of applying dye containing microcapsules onto the surface of polymer composite materials was developed. PVA (polyvinyl acetate) glue was used as a binding water-based agent, the filler consisted of dye-filled microcapsules and microcapsules filled with color developer mixed in a ratio of 1:1 in aqueous suspension. Concentration of the filler was 0 – 50% by weight in a liquid state.
2. Concentration of the filler in the sample was determined after complete drying of the sample (Fig. 3).

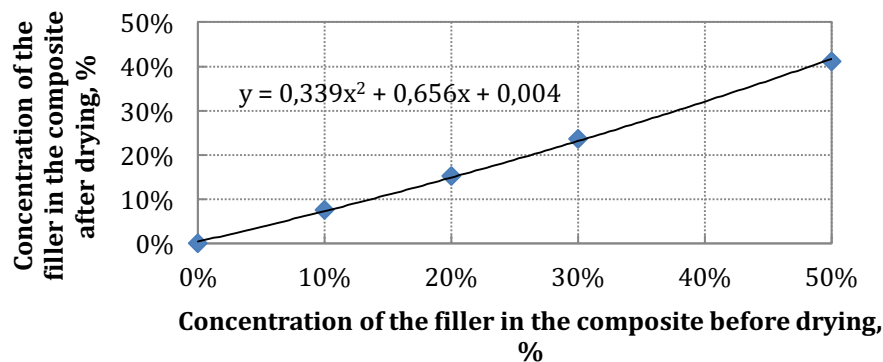


Fig. 3. Concentration of the filler in the composite after drying of water

3. Tension test of the samples was done (elastic modulus depending on concentration of the filler, stress-strain diagram). Staining of the samples using microcapsules was achieved on reaching the critical local deformation. The experiment demonstrated possibility of early diagnostics of destruction.

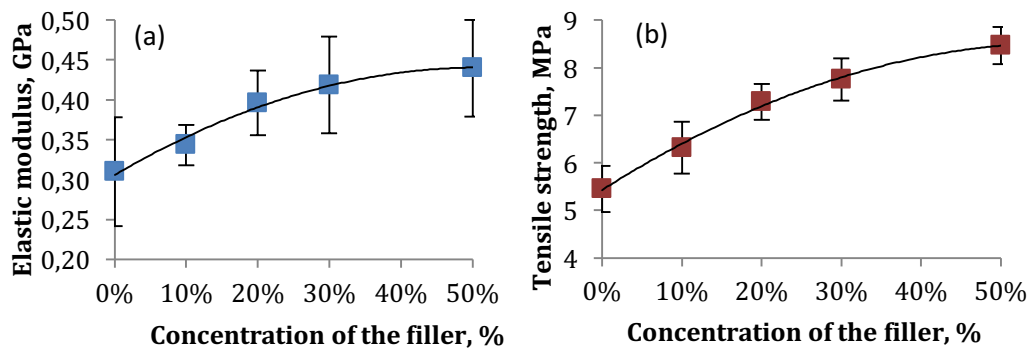


Fig. 4. (a) Elastic modulus of the samples depending of the filler concentration; (b) tensile strength of the samples depending of the filler concentration

4. Compression test of the polymer material with 2 types of shells of microcapsules was done with and without indenter. Stress-strain diagram was plotted, elastic modulus and compressive strength were determined.

The achieved results have the following scientific importance:

1. It has been demonstrated that organic substances leach into water from walls of polymeric pipes, however further studies are required in order to determine whether concentration of organic substances is sufficient to influence multiplication of aquatic microorganisms.

2. Possibility to develop a method for diagnostics of early destruction of polymer composite materials has been confirmed:
 - a) it is possible to observe destruction of the materials already at a stage of quasielastic deformation;
 - b) it is possible to adjust mechanical properties of dye containing microcapsules in order to visualise early destruction of polymer composite materials.

The achieved results of the 1st Period of the Project will promote development of methods of diagnostics of early destruction of surface of polymer composite materials during the next Periods of the Project.

2.4. Further research and practical exploitation of the results

(Describe further research activities that are planned, describe possibilities to practically exploit results)

Technological readiness has been achieved for the implementation of the 2nd Period of the Project. The achieved results demonstrate ability to continue implementation of the Project in accordance with the original application. **Therefore, the following two tasks are planned for the 2nd Period:**

1. Development of research methods for diagnostics of early destruction of surface of polymer composite materials: the method to research influence of aquatic microorganisms on early destruction of materials.
 - a) TOC leaching and microorganisms counting experiments will be performed using aging acceleration of polymeric pipes (at 60 °C temperature). Natural bacteria found in Evian water (these bacteria do not multiply without addition of the substrate unlike the bacteria present in tap water) and *E.Coli* bacteria will be used.
 - b) The research method for development of diagnostics of early destruction of surface of polymer composite materials using *in situ* electron emission spectroscopy will be developed (the Deliverable).
2. Development of research methods for diagnostics of early destruction of surface of polymer composite materials: the method to research visual recognition of early destruction using destruction-induced staining.
 - a) Damage indication coating changes mechanical properties of a composite material. Therefore it is necessary to take into account mechanical properties of the dye-containing microcapsules when designing constructions made from composite materials. By performing experimental measurements and simulations, mechanical properties of microcapsules embedded in an elastic matrix will be determined. Polymeric matrix with different concentration of microcapsules (0–50%) will be fabricated and tested. The obtained results will allow modelling mechanical behaviour of a matrix with embedded microcapsules.
 - b) The research method for development of diagnostics of early destruction of surface of polymer composite materials using destruction-induced staining will be developed (the Deliverable).

Practical exploitation of the achieved results:

Possibility to develop a method for diagnostics of early destruction of polymer composite materials has been confirmed. The experiments give evidence that the method will allow to observe the destruction already at a stage of quasielastic deformation. As a result:

- a) The knowledge about destruction processes taking place in polymer composite materials and water-supply polymeric pipes at nano and micro scale will be developed.
- b) Quality and safety of polymer composite materials and corresponding constructions, as well as sustainability and safety of water supply systems will be improved.

2.5. Dissemination and outreach activities

(Describe activities that were performed during reporting period to disseminate project results)

Conference abstract was submitted and accepted:

Aniskevich, A., Bulderberga, O, Dekhtyar, Yu., Denisova, V., Gruskevica, K., Juhna, T., Kozak, I., Romanova, M. Coloured Reactions and Emission of Electrons towards Early Diagnostics of Polymer Materials Overloading. 2nd International Conference „Innovative Materials, Structures and Technologies, September 30 – October 2, 2015, Riga, Latvia

Development of master theses was launched, the defence is in June, 2015:

1. Inguna Krista Anspoka. Influence of destruction of composite material on electron emission from composite material surface, supervisor Prof. A. Balodis
2. Irina Golovko. Influence of plastic water supply material on quality of drinking water, supervisor Assoc. Prof. K. Tihomirova

Development of bachelor theses was launched, the defence is in June, 2015:

1. Anna Korvena-Kosakovska. Early failure of composite material under mechanical load, supervisor Prof. A. Balodis
2. Ēriks Dombrovskis. Diagnostic method for early collapse of polymer pipes under mechanical load, supervisor Prof. J. Dehtjars
3. Toms Vāvere. Indirect determination of mechanical properties of polymer matrix spherical fillers, supervisors Dr. Sc. Ing. Andrejs Aņiskevičs, Msc. Olga Bulderberga

The doctoral thesis is being developed:

O. Bulderberga. Polymer composite with damage indication ability: development and determination of properties. Supervisor A. Aņiskevičs, the defence is planned in 2017.

Dissimination of results:

Project participants held three meetings to discuss the results of the Project (07.11.2014, 19.12.2014, 13.02.2015). The meetings were announced to all the staff of the departments involved in the implementation of the Project as well as for students who participated in the research developing their bachelor and master theses.

