"Nature works to maximum achievement at minimum effort. We have much to learn."
(http://www.cbid.gatech.edu/univ_labs.html)

A CASE STUDY ON NANOPARTICLES / NANOMATERIALS – SMALL THINGS BEHIND A STUNNING WORLD

by

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INTRODUCTION/BACKGROUND

What is "nano"? Well, without providing a definite answer to this question, nano is a popular (emerging) area of science and technology today. It has attracted the attention of researchers from all walks of life, from physics to chemistry to biology and engineering.

In today's scientific realm, the word nano describes physical lengthscales that are on the order of a billionth of a meter long. Nanoscale materials therefore lie in a physical size regime between bulk, macroscale, materials (the realm of condensed matter physics) and molecular compounds (the realm of traditional chemistry).

In this respect, nanoscale physics, chemistry, biology and engineering asks basic, yet unanswered, questions such as how the optical and electrical properties of a given material evolve from those of individual atoms or molecules to those of the parent bulk. Other questions that nanoscience asks include:

- How does one make a nanometer sized object?
- How do you make many (identical) nanometer sized objects?
- How do the optical and electrical properties of this nanoscale object change with size?
- How do its optical and electrical properties change with its "dimensionality"?
- How do charges behave in nanoscale objects?
- How does charge transport occur in these materials?
- Do these nanoscale materials posess new and previously undiscovered properties?
- Are they useful?

What are the relevant length scales for nano? Well, I guess it depends on who you talk to. On one hand some people call nano anything smaller than stuff on the micro level. This could mean dealing with stuff on the hundreds of nm scale. One useful perspective on a definition for the appropriate lengths scales for nano is a regime where the chemical, physical, optical and electrical properties of matter all become size and shape dependent.

In this problem-based learning activity, designed for non-science majors, students assume the role of scientists / teachers; their assignment is to work in team to design a laboratory experiment and put it in practice. Having in view that they know few things about nanoparticles / nanomaterials / nanotechnologies, were made available a range of information, experiments, based on which they can begin the laboratory essay.

NTSE database can provide to the students a basically and advanced approach on to the NANO area in terms of:

- Learning about nanoscale terms and concepts;
- Learning about crystal and nanocrystal structure;
- Learning about the process of obtaining nanoparticles;
- To learn about the way today's technology works;
- To link science lessons / axctivities / reseaches with the process of obtaining nanoparticles / nanocrystals;
- To learn the usage areas of nanotechnology in the context of nanoparticles;
- To design their own lesson plans in nano area field, as fitire teachers / professors.

PURPOSE

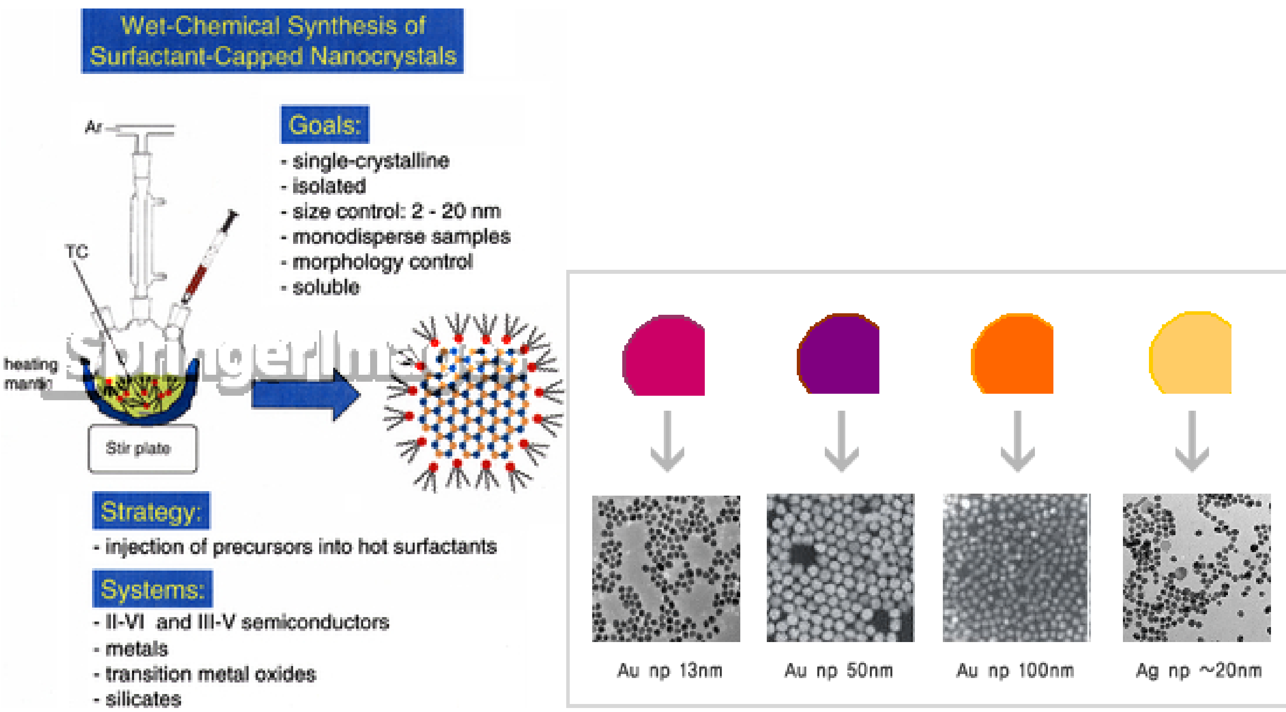
The purpose of the activity is to provide to the university students / prospective teachers a basically and advanced approach on to the NANO area. The students will develop an essay which must contain a virtual or real experiment that can be applied in laboratory. Having in view they are students from Science area (Physics-Chemistry) and moreover prospective teaches, they must take care also to didactical and pedagogical aspects. Some of the selected information / examples made available to the students are presented below, most of them beeing accessible from NTSE database (Repository) under L Education (General) and Q Science (General) sections. Here's the proposed list:

1. Obtaining of magnetite nanoparticles

Magnetite has an inverse spinel structure with oxygen forming a face-centered cubic crystal system. In magnetite, all tetrahedral sites are occupied by Fe^{3+} and octahedral sites are occupied by both Fe^{3+} and Fe^{2+}. Maghemite differs from magnetite in that all or most of the iron is in the trivalent state (Fe^{3+}) and by the presence of cation vacancies in the octahedral sites. Maghemite has a cubic unit cell in which each cell contains 32 O ions, 21⅓ Fe^{3+} ions and 2⅔ vacancies. The cations are distributed randomly over the 8 tetrahedral and 16 octahedral sites. Iron oxide nanoparticles are iron oxide particles with diameters between about 1 and 100 nanometers. The two main forms are magnetite (Fe_3O_4) and its oxidized form maghemite (γ-Fe_2O_3). They have attracted extensive interest due to their superparamagnetic properties and their potential applications in many fields (although Cu, Co and Ni are also highly magnetic materials, they are toxic and easily oxidized).

Applications of iron oxide nanoparticles include terabit magnetic storage devices, catalysis, sensors, and high-sensitivity biomolecular magnetic resonance imaging (MRI) for medical diagnosis and therapeutics. These applications require coating of the nanoparticles by agents such as long-chain fatty acids, alkyl-substituted amines and diols.

2. Colloidal synthesis of nanoparticles



Colloidal synthetic approaches have provided versatile tools for constructing uniform nanomaterials with controlled size, shape and crystalline phase. A variety of methods have been utilized to produce nanoparticles including milling, vapor-phase deposition techniques and solution-based synthesis. This section will be focused on colloidal solution-based methods as it is more commonly used in catalytic studies.

General colloidal synthesis primarily consists of three components: reactive precursors for particle formation, surfactants to direct particle size and shape, and solvents to act as a reaction medium. The choice of precursors, surfactants and solvents depend on the material and morphology desired. Typical reaction pathways include thermal decomposition, chemical reduction or oxidation, precipitation, sol–gel and galvanic exchange/replacement.

For metal nanoparticles, thermal decomposition is often chosen because it can produce small, spherical particles that are monodisperse, having size distributions within 5% ($\sigma_r \leq 5\%$). Precursors consist of zero-valent organometallics that are rapidly injected into hot high-boiling solvents with stabilizing surfactants. These reactions are often conducted using standard air-free techniques, as many of the precursors typically used are toxic and/or pyrophoric. Thermal reactions are often conducted at temperatures between 120 °C and 300 °C. Figure 1 shows a typical reaction setup consisting of a stir-bar, stir plate, heating mantle or oil bath, 3-neck round bottom flask fitted with a septum for injections, a thermocouple and a reflux condenser affixed to an inert gas line and bubbler.

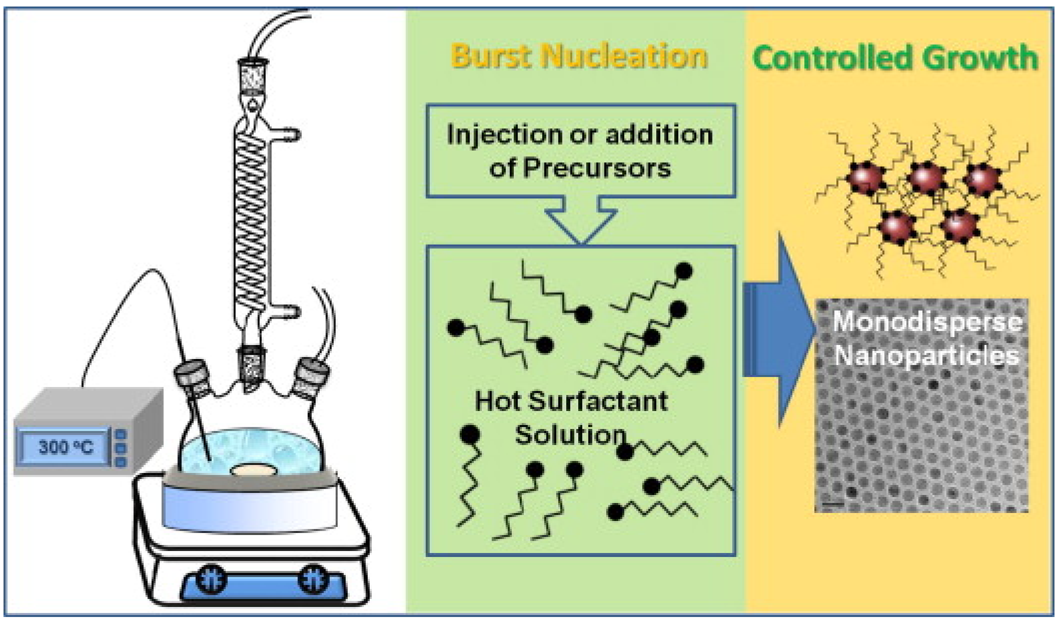


Figure 1. A typical reaction setup and concept for colloidal synthesis of nanoparticles.

Many metal chalcogenides are also made similarly with either the chalcogenide or metal precursor pre-dissolved in the reaction solution before injection. When using ionic precursors, reducing agents may also be needed to produce zero-valent metals or alloys. This allows for the formation of nanoparticles at ambient temperatures or in aqueous solutions. For slow growth, mild reducing agents are used, such as carboxylic acids or thermally activated 1,2-alkanediols. Stronger reducing agents, such as sodium borohydride or superhydride, are required for fast nucleation processes or for metal complexes with very negative reduction potentials.

3. Preparation of nanocatalysts: 2-D and 3-D catalysts

Colloidal metal nanoparticles can be applied to two types of catalysts; 2-dimensional (2-D) and 3-dimensional (3-D) catalysts. For decades, single crystals have been used for model studies of surfaces and catalysis, both as metal films and supports for metal particles. Similarly, 2-D catalysts are prepared by self-assembled nanoparticles deposited on a substrate by using the Langmuir–Blodgett technique (Figure 2). Surfactant stabilized colloidal nanoparticles floated on poor solvent – like water for the case of hydrophobic particles – then, assembled to form a closed packed array and deposited on a substrate by emersing the substrate form the liquid.

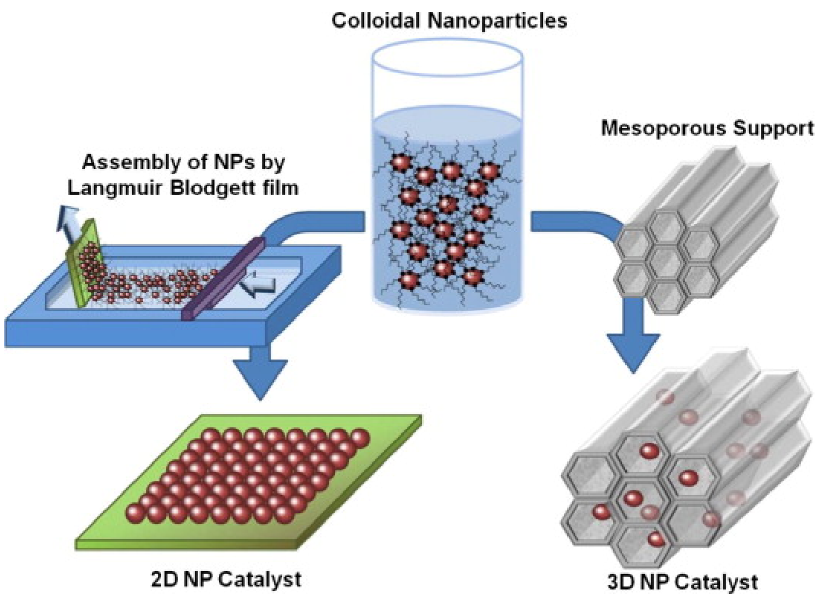


Figure 2. Schematic illustrations for preparation of colloidal nanoparticle-based 2D and 3D catalysts.

Conventional industrial catalysts are composed of metal nanoparticles supported on the high surface materials. In order to prepare for industrial heterogeneous catalysts with high surface area, two kinds of methods are mainly adopted: ion-exchange and incipient wetness. In both cases, active metal nanoparticles with a size range of 1–10 nm are deposited on and into metal oxides or carbons with high surface area. For ion-exchange, electrostatic interactions between metal precursor and support guarantee high dispersion of nanopaticles, whereas the incipient wetness provides a simple way to obtain nanoparticle catalysts in a large scale by using capillary force to load metal precursors in solution. However, both methods have a broad size distribution of nanoparticles, because of difficulties controlling thermal activation and reduction during the formation of particles on the supports.

4. Synthesis of nanomaterials

There are two approaches to the synthesis of nanomaterials and the fabrication of nanostructures: top-down and bottom-up. Attrition or milling is a typical top-down method in making nanoparticles, whereas the colloidal dispersion is a good example of bottom-up approach in the synthesis of nanoparticles. Lithography may be considered as a hybrid approach, since the growth of thin films is bottom-up whereas etching is top-down, while nanolithography and nano manipulation are commonly a bottom-up approach.

Both approaches play very important role in nanotechnology. There are advantages and disadvantages in both approaches. Among others, the biggest problem with top-down approach is the imperfection of the surface structure. It is well known that the conventional top-down techniques such as lithography can cause significant crystallographic damage to the processed and additional defects may be introduced even during the etching steps. For example, nanowires made by lithography are not smooth and may contain a lot of impurities and structural defects on surface. Such imperfections would have a significant impact on physical properties and surface chemistry of nanostructures and nanomaterials, since the surface over volume ratio in nanostructures and nanomaterials is very large. The surface imperfection would result in a reduced conductivity due to inelastic surface scattering, which in turn would lead to the generation of excessive heat and thus impose extra challenges to the device design and fabrication. Regardless of the surface imperfections and other defects that top-down approaches may introduce, they will continue to play an important role in the synthesis and fabrication of nanostructures and nanomaterials.

Bottom-up approach is often emphasized in nanotechnology literature, though bottom-up is nothing new in materials synthesis. Typical material synthesis is to build atom by atom on a very large scale, and has been in industrial use for over a century. Examples include the production of salt and nitrate in chemical industry, the growth of single crystals and deposition of films in electronic industry. For most materials, there is no difference in physical properties of materials regardless of the synthesis routes, provided that chemical composition, crystallinity, and microstructure of the material in question are identical.

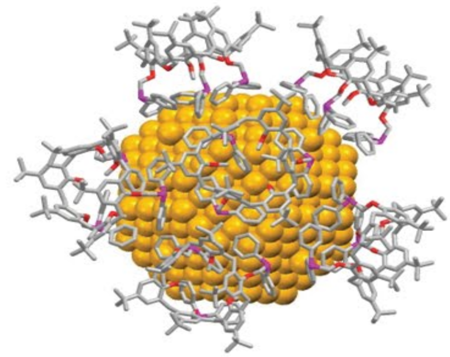
Of course, different synthesis and processing approaches often result in appreciable differences in chemical composition, crystallinity, and microstructure of the material due to kinetic reasons. Consequently, the material exhibits different physical properties.

Bottom-up approach refers to the build-up of a material from the bottom: atom-by-atom, molecule-by-molecule, or cluster-by-cluster. In organic chemistry and/or polymer science, we know polymers are synthesized by connecting individual monomers together.

In crystal growth, growth species, such as atoms, ions and molecules, after impinging onto the growth surface, assemble into crystal structure one after another.

Although the bottom-up approach is nothing new, it plays an important role in the fabrication and processing of nanostructures and nanomaterials.

5. Synthesis of metallic nanoparticles



The term metal nanoparticle is used to describe nano sized metals with dimensions (length, width or thickness) within the size range 1-100 nm. Metallic nanoparticles display properties that are quite different from those of individual atoms, surfaces or bulk materials. The main characteristics of MNPs are large surface area to volume ratio as compared to the bulk equivalents, large surface energies, existence as a transition between molecular and metallic states providing specific electronic structure (local density of states LDOS), have plasmon excitation, quantum confinement, short range ordering, increased number of kinks, contain a large number of low-coordination sites such as corners and edges, having a large number of ˝dangling bonds˝ and consequently specific and chemical properties and the ability to store excess electrons.

Their potential applications include, for example, use in biochemistry, in catalysis and as chemical and biological sensors, as systems for nanoelectronics and nanostructured magnetism.

Synthesis

Chemical methods Include chemical reduction of metal salts, alcohol reduction process, polyol process, microemulsions, thermal decomposition of metal salts and electrochemical synthesis.

Physical methods include exploding wire technique, Plasma, chemical vapour deposition, microwave irradiation, pulsed laser ablation, supercritical fluids, sono-chemical reduction and gamma radiation.

Reduction of metal complexes in dilute solutions is the general method of synthesis of metal colloidal dispersions, and a variety of methods have been developed to initiate and control the reduction reactions. In most cases the formation of monosized metallic nanoparticles is achieved by a combination of a low concentration of solute and polymeric monolayer adhering onto the growth surfaces. Both a low concentration and a polymeric monolayer can hinder the diffusion of growth species from the surrounding solution to the growth surfaces and the diffusion process is likely to be the rate limiting step of subsequent growth of initial nuclei, resulting in the formation of uniformly sized nanoparticles.

In the synthesis of metallic nanoparticles, or more specifically, metallic colloidal dispersion, various types of precursors, reduction reagents, other chemicals, and methods are used to promote or control the reduction reactions, the initial nucleation and the subsequent growth of initial nuclei.

The precursors include: elemental metals, inorganic salts and metal complexes, such as, Ni, Co, HAuC14, H,PtCl and PdCI2. Reduction reagents includes: sodium citrate, hydrogen peroxide, hydroxylamine hydrochloride, citric acid, carbon monoxide, phosphorus, hydrogen, formaldehyde, aqueous methanol, sodium carbonate and sodium hydroxide.

Metallic nanoparticles can also be prepared by an electrochemical deposition method employing a simple electrochemical cell containing only a metal anode and a metal or glassy carbon cathode. The electrolyte consists of organic solutions of tetra alkyl ammonium halogenides, which also serve as stabilizers for the produced metal nanoparticles. Upon application of an electric field, the anode undergoes oxidative dissolution forming metal ions, which would migrate toward the cathode. The reduction of metal ions by ammonium ions leads to the nucleation and subsequent growth of metallic nanoparticles in the solution.

OBJECTIVES

In completing the activity, students develop a variety of process skills critical to their further specialization including:

- working collaboratively;
- interpreting and prioritizing data / information;
- acquiring (by students) of investigative capacities and skills;
- defending an argument;
- increasing the students' confidence and their self-esteem to be involved in the discussion of scientific issues that can be found in ordinary and specialized newspapers;
- experimenting and obtaining reliable results.

From the scientific point of view, these activities allow students to learn about the defined characteristics of:

- nanotechnologies phenomena;
- possibilities of practical application related to theoretical knowledge of nanotechnologies;
- phenomena in the field of nanotechnologies;
- physical characteristics of the phenomena in the field of nanotechnologies;
- causal existed relations;
- carrying out the physical phenomena specific to nanotechnologies;
- application of the knowledge gained through the study of Science in related fields;
- presentation of the results of an investigative approach using specific Science terminology;
- advantages and disadvantages of nanotechnologies from the environmental perspective.

The activity designed for the *Physics-Chemistry university students (second year)*, sought to develop the knowledge, skills and attitudes of students involved, so that they can participate effectively in discussions on topical issues. At the same time, it stressed the direct exchanges of ideas and experience, laboratory work, to take a clearer picture of the investigative approach in action.

LEARNING RESULTS

When acquiring the information and after realizing the activities, students are able to:

- edit a report / essay where to submit arguments in respect of decisions taken and the related reasons;
- identify the consequences of applying nanotechnologies to human health, environment and society;
- retrieve specific information in the proposed websites;
- analyze selected information in relation to the proposed objectives;
- decide as a team how to deliver structured information in terms of didactical and pedagogical issues (terms, notions, experimental stages);
- analyze the pros and cons of applied nanotechnologies;
- submit collective conclusions made in front of the colleagues, in the laboratory.

CLASSROOM MANAGEMENT & SEQUENCE OF EVENTS

Before the activity starts, students should understand that many materials that belong to the natural world have properties which are the result of inherent nanostructures. The interaction of light, water and other materials with such nanostructures gives natural materials special properties that we can see with your own eyes. Nanomaterials study shows an increased interest in scientific research in recent decades. These materials, which are characterized by very small particle size, have great potential for use in many industrial applications, biomedical, electronics, etc. Alternatively, the instructor / teacher could present them some of the basic concepts concerning "nano" term and experimental examples by using appropriated images and videos.

The students from each team must share interesting facts and ideas found in the documented text, in order to arrive at a better understanding of the analyzed documents. It is recommended to start the documentation from the experiments proposed in the *Virtual Lab* of the *NTSE Project* (http://vlab.ntse-nanotech.eu/NanoVirtualLab/); the documentation must be made also using the *NTSE Repository* (http://ntse.ssai.valahia.ro/).

As a guideline for students this activity was divided into six "sessions" (see Table 1), some inside laboratory and some outside laboratory.

Table 1. Sequence of Events

Sequence	*Location*	*Activities*
Introduction and Session 1	Chemistry lab (50-90 min)	The teacher / instructor facilitates students discussion about nano terms. Students split into work teams (7 work teams formed by 4-5 students) and receive scenario / task and data and brainstorm potential main experiment to be maid
Session 2	Outside lab	Teams asses information and search for additional information
Session 3	Outside lab	Teams outline and produce drafts of essays
Session 4	Chemistry lab (30-40 min)	Teams present preliminary drafts to the teacher and verify for reagents, materials etc. in order to be available for experimental work
Session 5	Outside lab	Teams revise products according to reviewers' suggestions
Session 6	Chemistry lab (90 min)	Teams present products to entire group Each team reviews and evaluate one other team's designed experiment. The experiment selected (in terms of structure, didactical approach, scientific notions etc.) is made by all teams

RESOURCES

Procedural resources:

- methods and processes: experimenting, explanation, observation,, conversation, deliberation, discussion;
- form of organization: teams / groups, individual (frontal).

Material resources: video-projector, flipcharts, media texts, PCs, Internet, Colloidal Chemistry laboratory (instruments, reagents, utilities etc.)

PROCESS FINALIZATION

For finalizing the didactic process the university students from each team must express their personal opinions, answering to the following specific issues:

- Pedagogical approaches:
    - General pedagogical criteria:
        - ✓ The clarity of stated educational objectives and the expected results;

        - ✓ If the teaching materials meet the stated educational purpose;
        - ✓ The clarity of the learning objectives;
        - ✓ Tasks are clearly described;
        - ✓ If the activities are adapted to the target group.
    - Pedagogical requirements focused on teacher:
        - ✓ To set their own learning goals;
        - ✓ To search and explore information;
        - ✓ To collect and retrieve information;
        - ✓ Communicate with students;
        - ✓ To seek and receive support from experts in Nanotechnologies.
- Effectiveness of the content:
    - Information:
        - ✓ Information included are detailed and comprehensive;
        - ✓ Information included is relevant to the educational objectives set;
        - ✓ Information included are appropriate for the target group;
        - ✓ Information included helping to enrich the curriculum content;
        - ✓ Included information are related to relevant online resources;
        - ✓ Information included not contain labels or stereotypes political invoice / cultural / social / racial humiliation;
        - ✓ Information included are updated with current topics in the field of nanotechnology
        - ✓ Information sources are detailed.
    - Structure:
        - ✓ Information included are well structured and organized;
        - ✓ Included texts are well structured;
        - ✓ Labels are suitable and representative sections for the information;
        - ✓ Online resources related with information are relevant;
    - Presentation / Design:
        - ✓ Images and sounds included are properly referenced;
        - ✓ The texts are readable in terms of color, size, font type, arrangement and visual effects;
        - ✓ Graphics, images and videos included are well presented in terms of resolution, color and size;
        - ✓ Graphics, images, sound and video resources used are appropriate for the purpose;
        - ✓ Using images, videos and audio facilitates understanding;
    - Accuracy:
        - ✓ Links included in the proposed resources are appropriate and functional;

- ✓ The language used is correct syntactically and grammatically;
- ✓ Do you think there is a step that was omitted in the description of instructions or training materials?;

- Designed tasks:
  - ✓ The information provided was relevant and motivating for understanding topic task;
  - ✓ Do you consider appropriate / interesting the introduction of laboratory work related to nanomaterials / nanotechnologies?;
  - ✓ You find it useful to acquire additional knowledge about nanomaterials / nanotechnologies?

Important: the last part of the activity becomes very important, due to the fact that it represents a "debriefing" of everything the student has learned and lived during the whole process. The proposed questions dedicated to students substantiate in fact, the activity objectives.

ASSESSMENT SUGESTIONS

When the students make their final presentations has taken into consideration each aspect of their essay and also afterwards the laboratory work. Students receive a grade for this activity based on a combination of group score and an individual score. The individual score has been drawn from peer evaluations of the group process and evaluations of research notes collected by each student.

The evaluation of the students has to take into consideration the following items:

- ➢ understanding of the proposed / introduced concepts and terms;
- ➢ quality of retrieved information and investigation;
- ➢ clarity in the presentation of the selected information;
- ➢ active participation in various stages of the activity (power of argumentation, justification of presented opinions);
- ➢ laboratory work (abilities, skills, accuracy).

IMPACT ON STUDENTS

- ➢ using their real life perception and own life-experience as children / teenagers (especially linked to their knowledge);
- ➢ acquiring the necessary skills and capacities in their dual quality: as students and prospective teachers;
- ➢ maximizing the level of their involvement in the proposed topic;
- ➢ being eager to express their own opinions related to topic and to present their findings within the team and to the whole collective;
- ➢ being capable of gaining a deeper understanding of the current socio-scientific issues;
- ➢ better communicating with teacher and colleagues.

STUDENTS' FEEDBACK

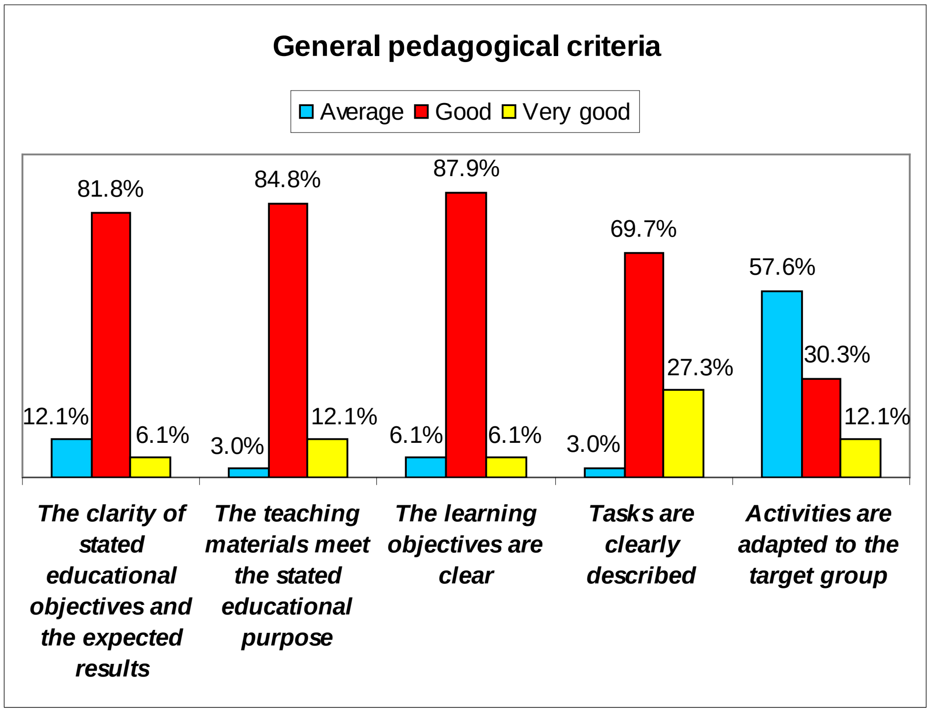
Expressed feedback:

- real and actual issues have been discussed;
- opportunity to work in groups and know better the colleagues;
- possibility to express own opinion on certain issues;
- possibility to communicate without fear with the colleagues, and also with the teacher;
- proper frame to argument the own opinions, as well as listening patiently to others;
- opportunity to compile documents and find out things that otherwise are not so easy to know.

All students declared that they learn something new, interesting and actual during the activity. They were motivated and very interested to the presented / discussed subjects due to the fact that those topics aren't greatly deepened during university curricula.

Processed feedback (graphical results):

Students were invited to fill in a questionnaire, in order to design a graphical feedback after processing their answers. *"Questionnaire for university students / prospective science teachers*" aimed at assessing and collecting information and suggestions on teaching effectiveness, content and usability of educational materials dedicated to teaching / learning Nanotechnologies created in the project Nano-Tech Science Education. The main sections of the questionnaire aimed pedagogical approaches and effectiveness of the content. The results are illustrated in the following diagrams.


General pedagogical criteria
Average
Good
Very good
81.8%
84.8%
87.9%
69.7%
57.6%
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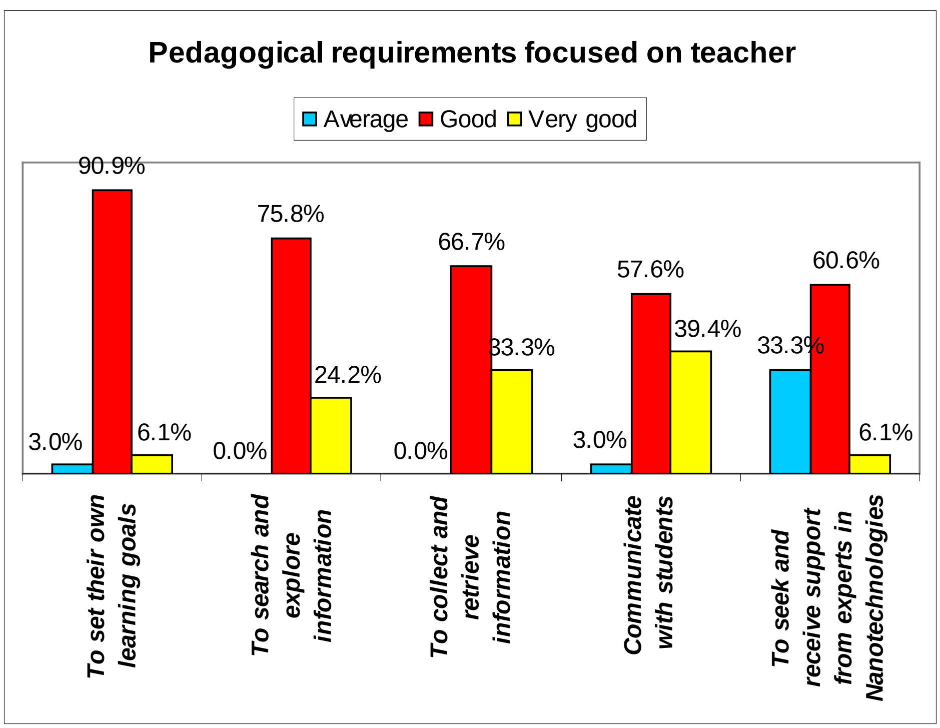
The clarity of stated educational objectives and the expected results
The teaching materials meet the stated educational purpose
The learning objectives are clear
Tasks are clearly described
Activities are adapted to the target group


Pedagogical requirements focused on teacher
Average
Good
Very good
90.9%
75.8%
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To set their own learning goals
To search and explore information
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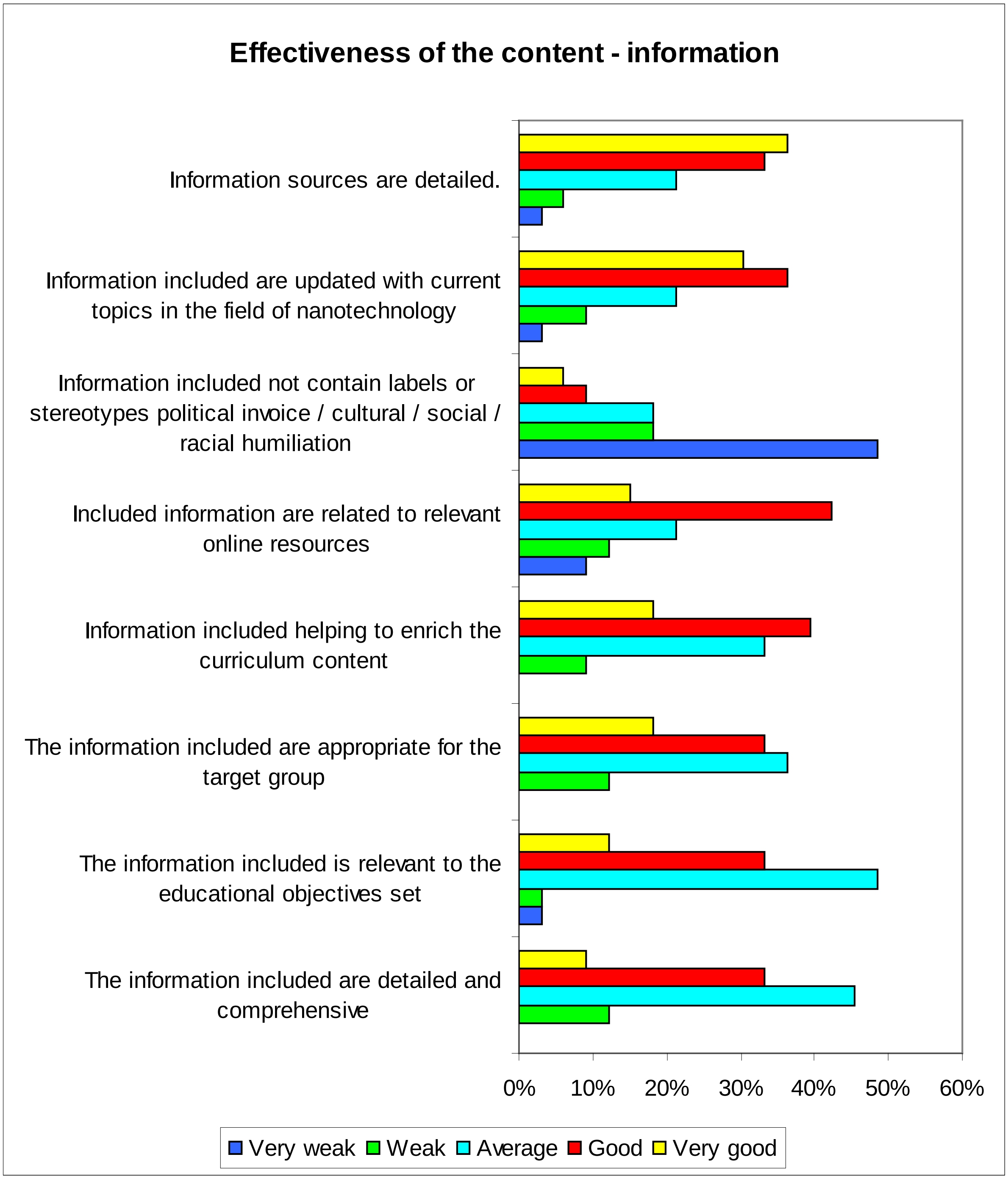

Effectiveness of the content - information
Information sources are detailed.
Information included are updated with current topics in the field of nanotechnology
Information included not contain labels or stereotypes political invoice / cultural / social / racial humiliation
Included information are related to relevant online resources
Information included helping to enrich the curriculum content
The information included are appropriate for the target group
The information included is relevant to the educational objectives set
The information included are detailed and comprehensive
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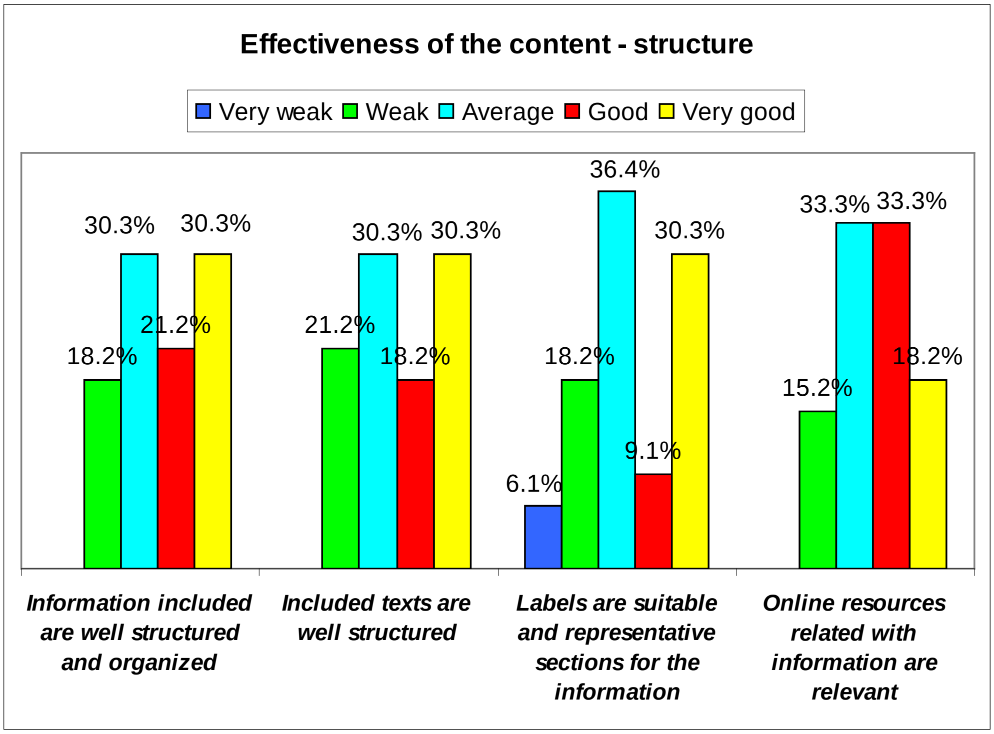
Weak
Average
Good
Very good


Effectiveness of the content - structure
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Weak
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Very good
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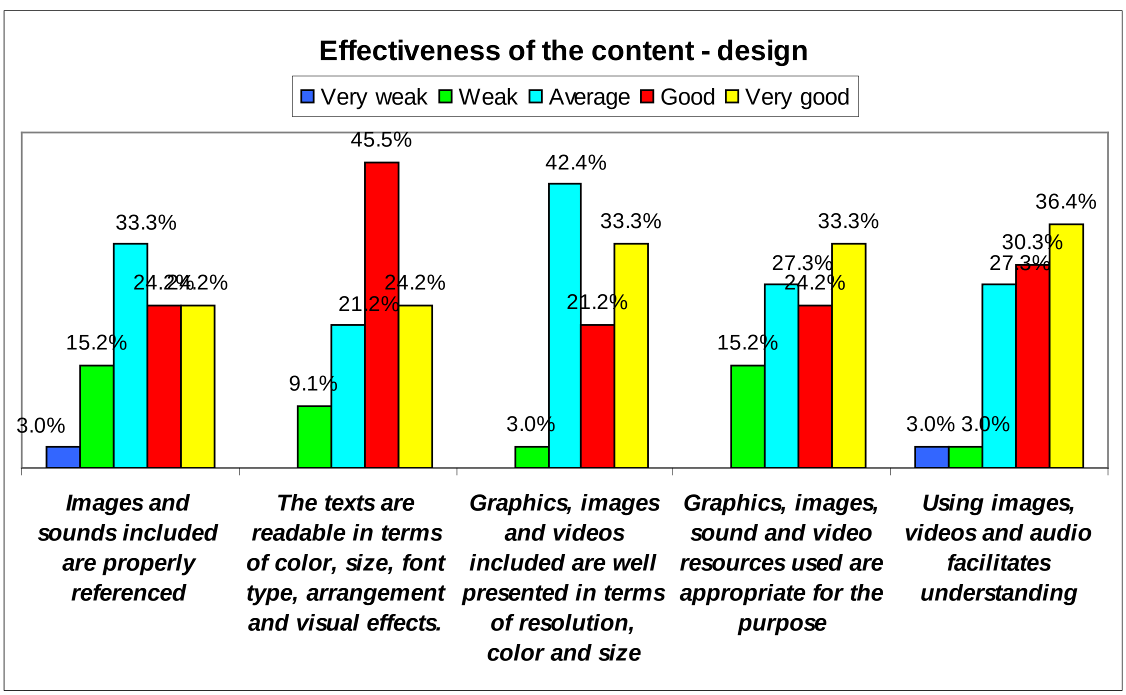
Information included are well structured and organized
Included texts are well structured
Labels are suitable and representative sections for the information
Online resources related with information are relevant


Effectiveness of the content - design
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Images and sounds included are properly referenced
The texts are readable in terms of color, size, font type, arrangement and visual effects.
Graphics, images and videos included are well presented in terms of resolution, color and size
Graphics, images, sound and video resources used are appropriate for the purpose
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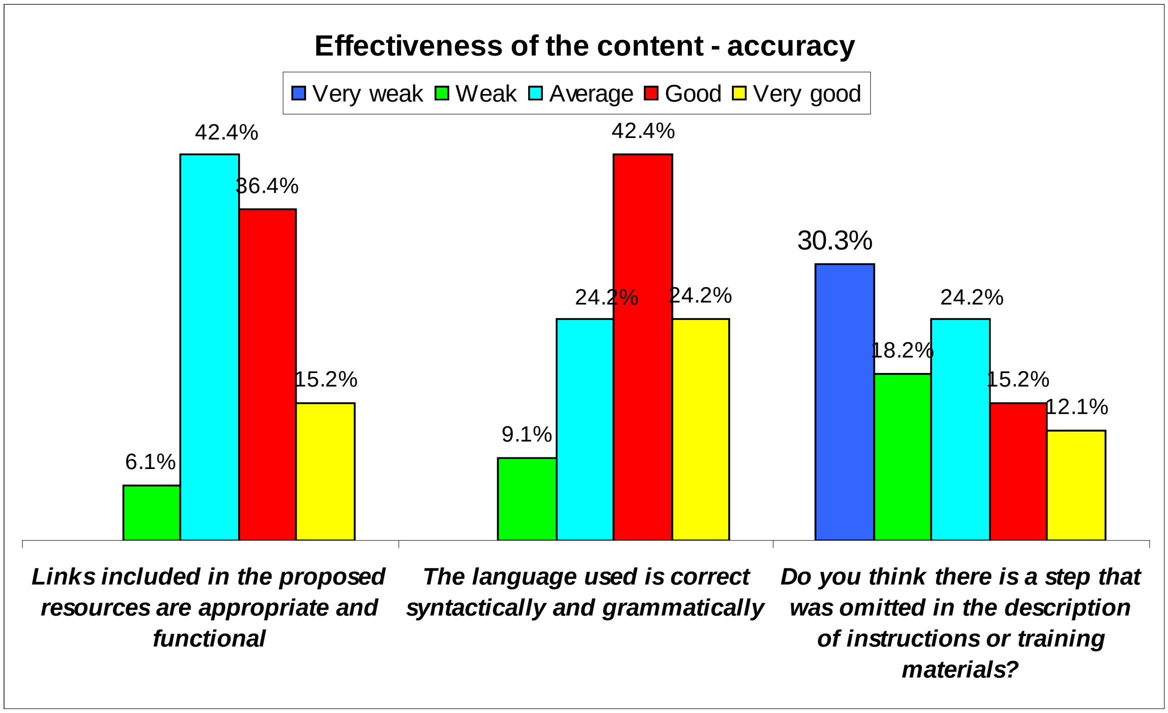
Effectiveness of the content - accuracy
Very weak
Weak
Average
Good
Very good
42.4%
36.4%
15.2%
6.1%
42.4%
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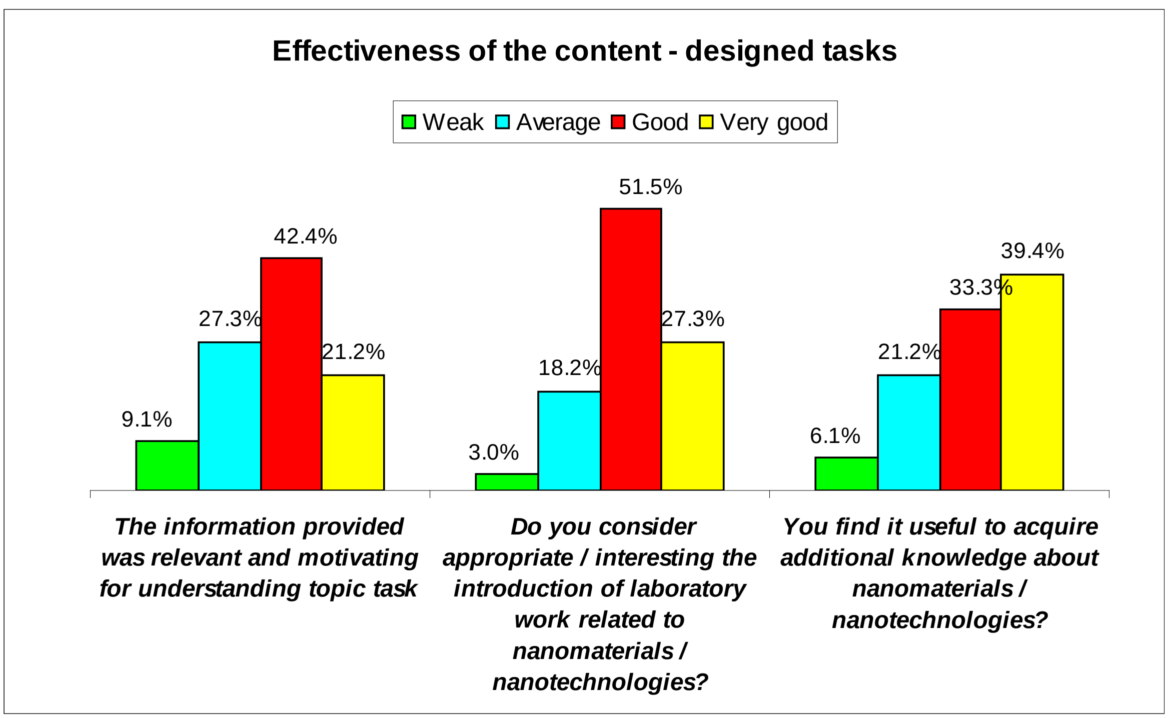
9.1%
30.3%
18.2%
24.2%
15.2%
12.1%
Links included in the proposed resources are appropriate and functional
The language used is correct syntactically and grammatically
Do you think there is a step that was omitted in the description of instructions or training materials?


Effectiveness of the content - designed tasks
Weak
Average
Good
Very good
9.1%
27.3%
42.4%
21.2%
3.0%
18.2%
51.5%
27.3%
6.1%
21.2%
33.3%
39.4%
The information provided was relevant and motivating for understanding topic task
Do you consider appropriate / interesting the introduction of laboratory work related to nanomaterials / nanotechnologies?
You find it useful to acquire additional knowledge about nanomaterials / nanotechnologies?

CONCLUSION

During the proposed students' laboratory activity, they were asked to effectively participate in all the laboratory activity stages, through experimenting, discussing, direct exchanging of ideas and concluding. From the university teacher's point of view, the objectives of the laboratory activity were achieved, not just taking into account the scientific point of view related to the presence of nanotechnology in the related activities, but also from the didactical / pedagogical perspective, contributing to the formation of the students, in order to make them to discern and to take responsible decisions, as future teachers / professors / researchers.

References (web, videos, documents)

1. http://vlab.ntse-nanotech.eu/NanoVirtualLab/experimentroom/908f4cedc98349d0b57e781ae3ea29c4
2. http://vlab.ntse-nanotech.eu/NanoVirtualLab/experimentroom/908f4cedc98349d0b57e781ae3ea29c1
3. http://vlab.ntse-nanotech.eu/NanoVirtualLab/experimentroom/908f4cedc98349d0b57e781ae3ea29c5
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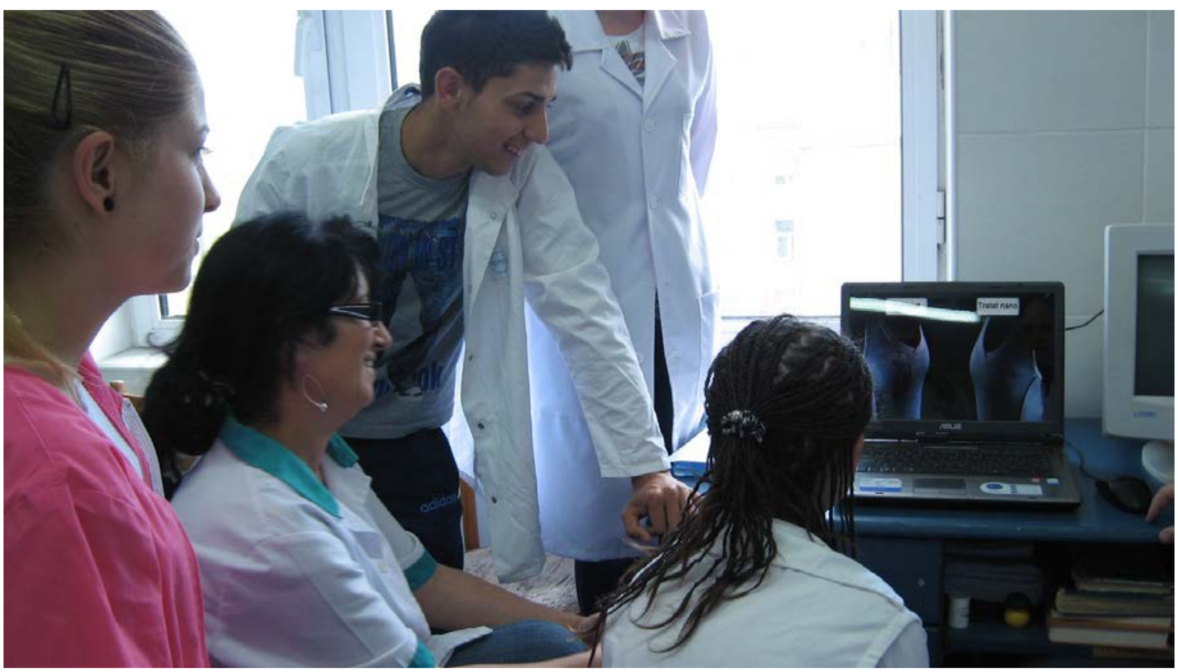
Images taken during the laboratory activity with university students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