

Small Materials to Solve Big Problems: Nanochemistry in Innovative Technology

Tutor Guide

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Table of Contents

Resource Overview.....	1
Flexibility within Resource.....	3
Module Learning Outcomes.....	3
Transferable skills.....	3
Assessment.....	4
Bibliography.....	5
Class organisation.....	6
Workshop 1: Module Induction and Information Retrieval Session.....	7
Workshop 2: Applications of Nanomaterials in Society.....	10
Workshop 3: Debate: 'Advantages and disadvantages of nanomaterials in society'.....	11
Workshop 4: Synthesis and cost of nanomaterials.....	14
Workshop 5: Introduction to Instrumentation.....	16
Workshop 6: Close of Module.....	18
Appendices.....	20
Appendix 1: Wiki guidelines.....	20
Appendix 2: Sample Gantt chart.....	27
Appendix 3: Student wiki/presentation checklist.....	28
Appendix 4: Guidelines for student reflective piece.....	29
Appendix 5: Case Studies.....	30
Case study 1: Mediwraps.....	31
Case study 2: Goldglow.....	37
Case study 3: Kleeenview.....	41
Case study 4: Nanoklenz.....	45
Case study 5: Solarsunrayz.....	56
Appendix 6: Synthetic procedures for preparation of nanomaterials.....	60
Appendix 7: Full length urls.....	70
References.....	71

Resource Overview

This resource is designed for year one or two of an undergraduate BSc honours chemistry or physical sciences course. The context of the resource is to examine nanomaterials for the purpose of preparing a commercial product, while giving the students an introduction to the synthesis and characterisation of nanomaterials. Context/Problem Based Learning (C/PBL) is a teaching methodology that aims to increase students' engagement with a subject by delivering courses based upon real-life applications of the principles, techniques and experiments students encounter in their undergraduate courses. These real world contexts are presented in the form of problem scenarios which are ill-defined, and have a number of satisfactory solutions. Learners work collaboratively to solve problems and acquire new knowledge and present the outcomes or product. This approach provides the opportunity to develop valuable transferable skills such as communication, team working and problem solving. Students are encouraged to take control of their learning and real world examples are used as an effective means to promote real learning. Academic staff adopt the role of facilitator or guide during this process. It is recommended that the following review on context and problem based learning be consulted for further information; T.L. Overton, Context and Problem-Based Learning, New Directions, Issue 3, Oct. 2007, pages 7-12.

The resource is a paper based exercise, but will be supported with data previously collected from experiments, which is available electronically. Students will be presented with an introduction to nanochemistry and with self directed exercises to introduce concepts. Characterisation techniques for nanomaterials showing the instrumentation used and the applications of these materials in a real life context will be considered. Figure 1 provides an outline of how this will be achieved. The module has been designed to allow students to build an introductory level knowledge about nanomaterials and their applications during the workshops, which include student presentations and debates as depicted in Figure 1. The case study will allow them as groups to build on their learning to compile their case study material into a wiki format and present it to the class. Introductory support on the synthesis of nanomaterials and the characterisation techniques involved have been supported with electronic resources provided. The tutor guide is also supported with an annotated bibliography for each case study so that the tutor may provide some papers of interest. The synthesis and safety data for some of the nanomaterials are also supplied in the appendix to the tutor guide should you wish to include a demonstration or 'hands on' practical.

The workshops have been designed to promote student driven learning. Each workshop should take up to three hours per session. Access to nearby computer facilities throughout the module would be ideal for facilitating the workshops. The staff student ratio recommended for each workshop would be one tutor per thirty students maximum. The assessment has been aligned with the learning outcomes (both for the module and the individual workshops) to address transferable skills in parallel with the scientific context.

The workshops will encompass: student presentations, debates, group wikis, and individual reflective statements.

Guidelines are provided in the student guide appendices on:

- Using a wiki
- Preparing presentations
- Writing a reflective piece
- Plagiarism

Students should be directed to read these and follow them when preparing work.

This resource aligns with the RSC Chemistry for Tomorrow's World priority areas "Future cities" and "Human health."

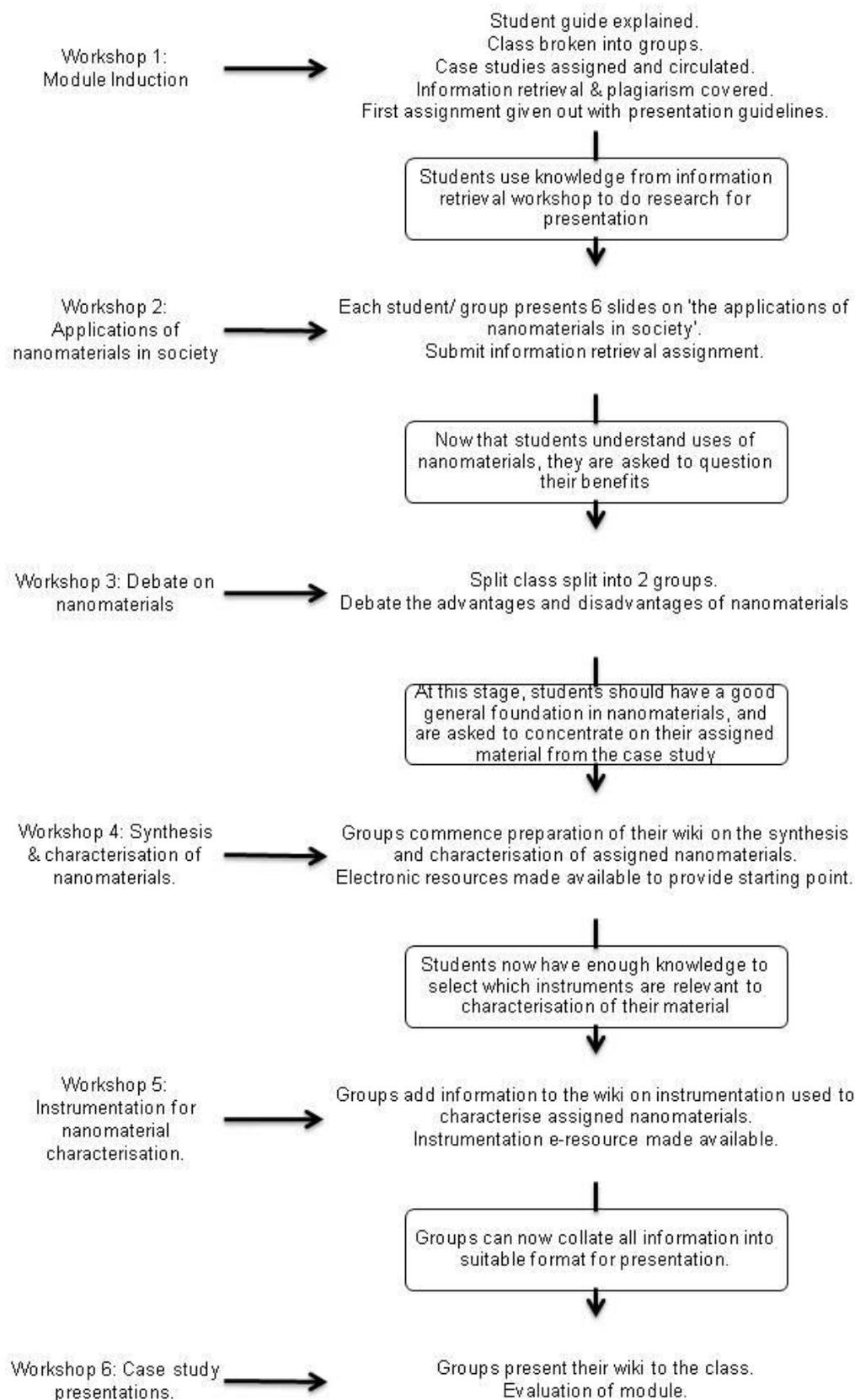


Figure 1: Suggested overview of workshop sessions

Flexibility within Resource

The resource may be altered to suit the needs of the class group. Where students already have an understanding of nanomaterials or the time available is short, workshops 4-6 can easily be run as a standalone resource. If the module tutor wishes to lengthen the module, or to incorporate laboratory sessions, detailed laboratory procedures are provided in Appendix 6.

There are many advantages to using a wiki when collaboration on a group project is required and these are dealt with in more detail in the Appendix. To summarise, it provides an effective and flexible means for learners to work as a team on a report or presentation while generating an archive of all information used and of all previous versions of the final pages. The main benefit to a tutor is that the quality and quantity of contributions made by each student can be tracked relatively easily and that the process as well as the product can be assessed.

Although it is recommended that a wiki be used as a component of this C/PBL activity, an alternative can be adopted if preferred. Some type of online interaction among a group such as a discussion board or online group is very useful, and, if this has been set up by the tutor, there is the advantage that they will be able to monitor progress being made. If this option is not used, the weekly group meeting summaries that are required from each group can be used to monitor progress and to check that all members are making a contribution. To provide a facility similar to the wiki for organisation of the work being undertaken, it would be useful for groups to use a ring binder with sections that correspond to the main parts of the report to which useful documents and draft work can be added. Under these circumstances, it is recommended that the students produce a final printed report with this draft work and supporting information is submitted as an appendix.

Module Learning Outcomes

On completion of the resource, the learner will be able to do the following within the context provided:

- Describe nanomaterials and the difference between their properties and those of the bulk material.
- Explain the applications of nanomaterials and discuss the advantages and disadvantages of using them.
- Identify instruments suitable for the characterisation of nanomaterials.
- Suggest which instruments and techniques will be suitable for the identification of particular nanomaterials.
- Prepare group reports on their research of the subject area and support their work with individual reflective statements.

Transferable skills

This resource allows the learner to further develop the following transferable skills:

- Problem solving: learners work in groups to address the brief presented in the contextualised scenario.
- Analytical and critical thinking: using their background knowledge to inform opinion in the debate, deciding on synthetic protocols and characterisation methods.
- Team work: learners work in groups to complete the task assigned, use a wiki to facilitate collaboration and meet between sessions to review progress.

- Communication skills: Learners present (oral presentation) and report (wiki) on the scientific work performed in keeping with the context.
- Independent learning: learners can justify decisions, assumptions and conclusions made with reference to supporting documents and literature in order to produce a logical and clearly reasoned scientific proposal.
- Information technology skills: learners use a wiki to collaborate and develop their ability to use word-processing, spreadsheet, presentation, chemical drawing and library database software.
- Metacognition: learners reflect on the process involved in working on the brief given, the extent to which the stated learning outcomes were met and to which their transferable skills were developed.

Assessment

Table 1 provides an outline for the assessment of this module. For certain workshops, additional detail is given on assessment of specific tasks driving the session, but in general, this can be used as a guideline for the module. A peer assessment form is provided in workshop 6 to give the students and tutor an opportunity to give immediate anonymous feedback during the presentations.

Table 1: Overall assessment of module

Assessment Component	Mark Allocation	Assessment mark
Information retrieval (Workshop 1)	Individual	10%
PowerPoint presentation (Workshop 2)	Individual	20%
Debate (Workshop 3)	Individual	20%
Case Study		50%
Breakdown:		
Attendance and contribution at workshop 4	Individual	(5%)
Attendance and contribution at workshop 5	Individual	(5%)
Attendance and contribution at workshop 6	Individual	(5%)
Group wiki	Group	(20%)
Peer assessment mark for wiki presentation	Group	(5%)
Final individual reflective piece	Individual	(10%)

Since this module is continuously assessed, the student should submit an assignment or present/prepare work each week by the deadlines shown in Table 2. It is very important that they submit assignments on or before the deadline, and that they check the timetable below carefully. All assignments should be prepared/submitted on a weekly basis as shown below during the workshop session unless otherwise indicated by you. It is expected that the tutor should aim to return material within a week so that feedback can be acted on.

The student will be required to submit an individual reflective piece on their engagement with the module, which will receive an individual mark. This should be submitted the week after the module has ended.

Table 2: Outline of module assessment submission/presentation dates

Module Week	Outline of Assessment Description
Week 2 Date:	Submit a library information assignment from week 1. Present a six slide presentation per individual on 'the applications of nanomaterials in society'.
Week 3 Date:	Submit debate points to module tutor in advance of the debate. Attend and participate in the debate.
Week 4 Date:	Prepare material for wiki on the preparation of nanomaterials and characterisation for case study.
Week 5 Date:	Prepare material for wiki on instrumentation for case study. Finalise material for the project description template.
Week 6 Date:	Present as a group the case study wiki and submit a compilation of individual work.

Bibliography

This bibliography is provided to students to give a starting point for their research. Some sources are of poorer quality than others to encourage students to use their judgement when choosing which sources of information to use.

Nanochemistry: A Chemical Approach to Nanomaterials, Ozin, G.A., Arsenault, A.C., Cademartiri, L.; 2009, Published by Royal Society of Chemistry

Concepts of Nanochemistry, Cademartiri, L., Ozin, G.A., Lehn, J.M.; 2009. Wiley-VCH.

Nanomaterials chemistry: recent developments and new directions, Müller, A. K. Cheetham, Wiley-VCH, 2007.

<http://tinyurl.com/nanochem1>, accessed August 2011.

First Experiences Teaching Experimental Nanoscale Science and Technology to Undergraduate Students, Shanov, V., Yun Yeo-Heung; Smith, L., Iyer, S.S., Jadhav, S., Hoang, T.B., Gorton, A., Schulz, M., Mantei, T., Abel, J., Barbour, C., D'Souza, N., Direnzi, C., Dlesk, D., Kier, Z., Negassi, N., Schaub, R., Seger, K., Wagner, R., Witt, E., Bickle, J., Paputsky, I., Gerner, F., IEEE-NANO 2006, 1, 410- 413.

Feynman's Fancy; P. Ball, in *Chemistry World*, Royal Society of Chemistry, 2009, vol. January, ch. 58, pp. 58-62. <http://tinyurl.com/nanochem2>

Semiconductor Clusters, Nanocrystals, and Quantum Dots. Alivisatos, A. P.; Science, February 1996, 271, 933 – 937

Quantum dot technologies explained in three levels of difficulty <http://tinyurl.com/nanochem3>

Resource for anyone interested in learning about Nanoscience and Nanotechnology
<http://tinyurl.com/nanochem4>

Putting the nano into nanochemistry, Chemistry World, December 2005 <http://tinyurl.com/nanochem5>

Nano-regulation creeps closer, Chemistry World, February 2009 <http://tinyurl.com/nanochem6>

Why do we worry about Nanomaterials?, Report by Royal Society of Chemistry Environmental Health and Safety Committee, August 2010 <http://tinyurl.com/nanochem7>

10 things you should know about nano <http://tinyurl.com/nanochem8>

A full suite of learning materials, including teacher and student materials, for introducing the topic of nanotechnology <http://tinyurl.com/nanochem9>

Video Lab Manual for Nanotechnology experiments <http://tinyurl.com/nanochem10>

Introduction to topics on Nanotech Project: <http://tinyurl.com/nanochem11>

Contextualising nanotechnology in chemistry education, O'Connor, C., Hayden, H.; *Chemistry Education Research and Practice*, 2008, 9, 35-42 (Accessible from the RSC website)

ACS Nanotation – collection of papers and discussions on nanotechnology <http://tinyurl.com/nanochem12>

Other useful links <http://tinyurl.com/nanochem13>

Nottingham Science – What is nanotechnology <http://tinyurl.com/nanochem14>

Online educational resources on nanotechnology <http://tinyurl.com/nanochem15>

TED^x Talk: Peter Janse van Rensburg - 10 Nifty Things You Can Do With Nanotechnology <http://tinyurl.com/nanochem16>

TED^x Talk: Dr. Wade Adams – Nanotechnology: Making Small Stuff do Big Things <http://tinyurl.com/nanochem17>

Wikipedia article on nanochemistry (last accessed April 2012) <http://tinyurl.com/nanochem41> and nanoparticles <http://tinyurl.com/nanochem42>

Research into nanotechnology to prevent AIDS by Jacob Silverman (last accessed April 2012) <http://tinyurl.com/nanochem43>

How could gold save my life? (last accessed April 2012) <http://tinyurl.com/nanochem44>

How Nanotechnology Works by Kevin Bonsor and Jonathan Strickland (last accessed April 2012) <http://tinyurl.com/nanochem45>

British scientists 'seek and destroy' cancer cells using iron nanoparticles By David Derbyshire, Daily Mail, 5 October 2009 (last accessed April 2012) <http://tinyurl.com/nanochem46>

“Atomic, Monatomic, Subatomic Particles” <http://tinyurl.com/nanochem47>

Class organisation

The students should be in groups of thirty (maximum) for workshops. Two tutors should be made available per group for the debate session in Workshop 3, and for the evaluation of Workshop 6.

Once the learners have been assigned into their groups by their tutor a table listing these groups can be inserted into page six of the student guide.

Workshop 1: Module Induction and Information Retrieval Session

Introduction to Case Study: Preparation and characterisation of a nanomaterial.

The tutor will give a module induction presentation (as provided) to the class, and circulate the student guide (or place on a virtual learning environment (VLE) prior to this session). The module induction should take approximately one hour. The tutor should also organise an information session from one of the college librarians, if the students have previously engaged with the library information systems a brief handout may be circulated as an alternative. This workshop should ideally be carried out in a computer room or the students should be allocated time to go to one.

The class should be split into groups, and given a contextualised case study under the three themes highlighted in Figure 2. Assign one of five case studies to a group. If there is a large number of groups, you may wish to subdivide the carbon allotrope case study by specifying which material they study, as shown, to give eight possible topics. Each case study specifies a nanomaterial to research within a particular context. The contexts should be addressed at each workshop over the course of the module.

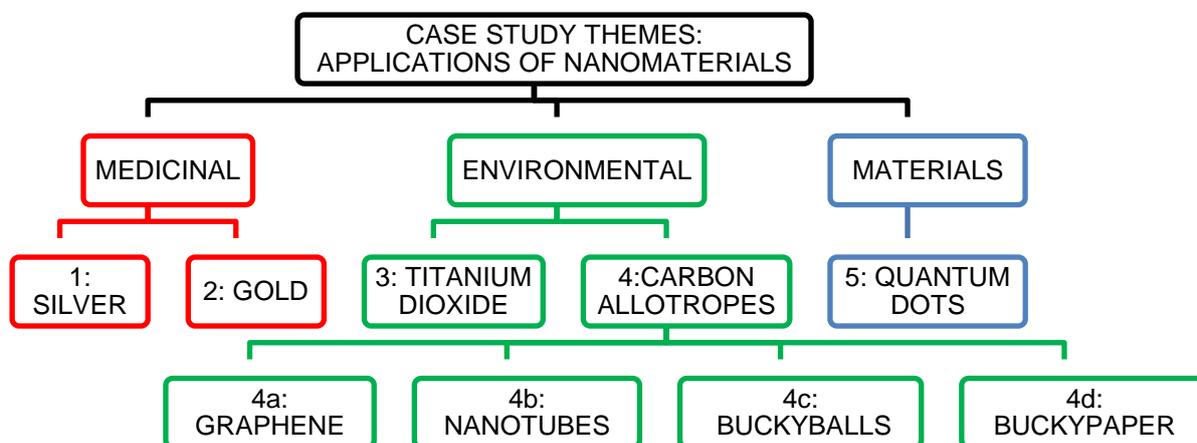


Figure 2: Themes for case study and examples of relevant nanomaterials.

As explained, there is a possibility of five to eight case studies as shown, however given the variety of content covered under carbon allotropes, this may be further sub-divided. Each case study has an associated scenario on which the group has to carry out research to address a real life application. They then prepare a group presentation/report in the form of a wiki (the content of which should follow a project description template as supplied). Each individual will also have to provide a reflective piece on their contribution to the case study and the final report.

As outlined in Table 1, assignments will be due each week, details of which will be provided in each workshop description that follows. A major output from the students work will take place in workshop 4 and 5, when they will be expected to create a group wiki on their case study on:

1. The synthesis and characterisation of their assigned nanomaterial and
2. Three types of analytical instruments they have selected to characterise their nanomaterial.

The students are provided with a template for the content of the wiki and a word count with each case study. Guidelines for the preparation of a wiki and an example are also given. Each workshop has been supported with an annotated bibliography. If the students require further guidance on getting their research started, they should consult their module tutor as soon as possible. Students are also required to submit an individual folder of work at the end of the module, this should be compiled as the module progresses as hardcopy evidence of the work they are doing. You can use this when assessing individual contributions to group activities.

Workshop 6 will be the culmination of the students work as a group. The case study wiki will be presented by the group to the class. The presentation will be assessed by the tutor and the student peers. A short individual reflective piece is also submitted which provides feedback on how individual students contributed to the case study and what they learned during the process of the group exercise and the module as a whole.

Upon completion of the case studies, the students will be able to:

- Provide an overview of the synthesis of the nanomaterial assigned.
- Create an information portal via a wiki/PowerPoint presentation with other members of their group.
- Construct a well designed description from the information retrieved.
- Provide an overview of an instrument capable of characterising nanomaterials.
- Evaluate the importance of the data provided from the instrument.
- Construct a well designed instrument description from the information retrieved.
- Evaluate the importance of the data provided from the literature.

Assignment to be completed during Workshop 1: Information Retrieval

The information retrieval session is best facilitated by a librarian from your institution. This session may take up to one hour. During the Library Resources/Information Retrieval session, you will need to introduce students to the web-based and library-based sources of information available to them in your institution such as Web of Knowledge and Google Scholar. These resources will prove very useful when researching the background information for other assignments. Upon completion of this session students are asked to reflect and build on their learning by completing a reflective exercise, and preparing a short PowerPoint presentation using the resources available from the Library Services.

Student Learning Objectives

The assignment will enable the student to:

- Demonstrate an awareness of the types of information resources available
- Summarise where to find them within a Library context
- Begin to evaluate the relevance and reliability of academic resources
- Apply them to a chemistry assignment

Assignment to be completed before Workshop 2

After completing the information retrieval workshop, the students must complete the following two exercises:

1. Using the library resources available, prepare a PowerPoint presentation for the next workshop on 'Applications of Nanomaterials in Society' under the theme of your case study. The slides should cover current applications of the relevant nanomaterials in society or research into potential uses, with relevant diagrams, images and references. The slides must be e-mailed to the module tutor by 5pm the day before the presentations, and a copy should be brought on memory key the day of the presentation. Tips on preparation of a PowerPoint presentation are provided in the appendix of the student guide to assist them in preparing a professional presentation.
2. The student can choose **one** of the following assignment formats to reflect on what they learned about information retrieval during workshop 1:

Outline the different types of academic information resources available for chemistry and where to find them **as a visual representation** e.g. a mindmap or flowchart.

They can use freeware such as:

- EDraw (<http://tinyurl.com/nanochem21>)
- Bubbl (<http://bubbl.us/>)
- VisualMind v11 (<http://tinyurl.com/nanochem22>)
- MS PowerPoint or other software

Please print out your mindmap for submission.

OR

Prepare a written reflective piece (approx 500 words typed) indicating:

- which information resource types they were shown in the class which were new to them
- what they need to access them
- where they can locate the different types of resources
- which parts of the session on information retrieval they found most useful

Workshop 2: Applications of Nanomaterials in Society

Assignment to be completed during Workshop 2

In this workshop the student group will present six PowerPoint slides on the applications of nanomaterials in society under the theme of their case study. The slides must be e-mailed to you by 5 pm the day before the presentations and a copy should be brought on memory key on the day of the presentation. Students should be reminded that they should use this opportunity to learn about other applications of nanomaterials from the other presenters and that they may also pick up some presentation skills from the other students.

Student Learning Objectives

- To retrieve relevant information about nanomaterials and their applications.
- To understand how to use PowerPoint to prepare a professional presentation.
- To evaluate and provide a well supported opinion on the application of nanomaterials in society.
- To participate in a discussion of academic interest with other members of their peer-group.
- To discuss the role of nanomaterials in society, and particularly its applications in society.
- To evaluate the importance of a number of applications of nanomaterials in society.

Assessment

Table 3: Assessment components for the presentation.

Assessment component	Mark
Submission of powerpoint presentation	30%
Presentation marks:	
Attendance at presentation	10%
Content	15%
Clarity and structure	10%
Visual impact	15%
References	10%
Questions answered at end of talk.	10%

Assignment to be completed before Workshop 3

Following on from their research on the applications of nanomaterials in society, the students should submit 10 debate points (5 advantages and 5 disadvantages in relation to the applications of nanomaterials in society). The points should be validated with a reference from a textbook, journal publication, newspaper or some other reliable source. They should submit the debate points to you prior to the debate taking place, and bring a copy with them on the day of the debate. The tutor should aim to return comments/ feedback at the start of Workshop 3 if possible or else give feedback to the group on the points submitted.

Workshop 3: Debate: 'Advantages and disadvantages of nanomaterials in society'.

Assignment to be completed during Workshop 3

On the day of the debate the class will be split into 2 groups and within each group a spokesperson and a recorder will be elected. Ideally the two groups would go to two separate rooms to prepare for the debate. The recorder will be required to take note of at least 10 points (for and against) from their group on a white board/flip chart. The group should agree on their top 10 points to give to the spokesperson for the debate.

The debate should be structured so that each spokesperson can state their points without interruption and once the points have been delivered from each side the group can be asked to contribute to the debate (usually better facilitated if the students raise their hand to make a point). It would be useful if you can summarise some of the main points on the board/flip chart to wrap up the debate session. If statements or claims are made that cannot be supported by examples or details, students can be reminded that evidence is important.

This assignment is assessed as an individual one; depending on the class size it may be done in pairs. The assignment will be marked on the submission of the debate points and the attendance and participation at the debate and the marking scheme is given in Table 4. The students will not be marked directly on winning or losing the debate.

Student Learning Objectives

- To evaluate and provide a well supported opinion on the application of nanomaterials in society.
- To participate in a discussion of academic interest with other members of their peer-group.
- To discuss the role of nanomaterials in society and particularly its applications in society.
- To construct a well-reasoned argument based on the information retrieved.

Tutor role in debate:

- Collect debate points prior to the debate session.
- Explain to students what is required from them in debate and discuss appropriate etiquette (e.g. showing respect and not interrupting each other).
- Inform students of workshop structure.
- Facilitate student input into debate.

Example of workshop structure

Before workshop 3 commences, make sure you have received a list of advantages and disadvantages from each student. If students have not submitted points they should be made aware that this will affect their overall mark.

In workshop:

1. Randomly split the class into two groups, and assign each group a side to argue for the debate.

2. Ask each group to nominate a spokesperson and recorder, within five minutes of the groups getting together to prevent time wasting.
3. Provide the students with a time limit to work within when preparing the group debate points (length of time will vary depending on group size but 10-15 minutes should be plenty). At the end of this time, they should have a list of 10 points prepared by collating points from all group members.
4. Bring groups back together. It may be useful to ask them to sit in their groups.
5. Each speaker presents their points without being interrupted by the other team.
6. When both groups have presented their points the floor can be opened to debate. You may wish to chair the debate, asking students to put their hand up to raise a point through you. You can then ask a member of the other team to volunteer to answer or select a student yourself that you suggest answers.
7. Try to record the main issues on the white board.
8. Towards the end of the debate, remind the group that those who haven't contributed will not get marked as highly as those who have to encourage everyone to get involved. (This may not be necessary).

Assessment

Table 4: Assessment components for the debate

Assessment Component	Assessment Mark
1. Submission of debate points	30%
2. Attendance and participation at debate:	
a. Attended the debate	10%
b. Attended the debate and gave points to the group	20%
c. Attended the debate and had input into the groups selection of points to make	30%
d. Attended the debate and had some input into the group discussion	40%
e. Attended the debate and had a lot of constructive involvement in the group discussion	50%
f. Attended the debate and had input from (b - e)	60%
g. Attended the debate and made a major contribution to the debate which was supported by research findings	70%

Note: It is useful to have two tutors available when assessing 2 b and c.

Example of debate points

Advantages:

1. Stronger lighter materials
2. Different properties than the bulk materials.
3. Wide range of applications.
4. Allows for miniaturization of technology.

Disadvantages:

1. Nanotoxicology – not enough known.
2. Very specialised equipment required to characterize the nanomaterials.
3. Difficulty in purifying nanomaterials.
4. Can be expensive to prepare nanomaterials.

Assignment to be completed before Workshop 4

In preparation for the next workshop, students should research synthetic methods for preparation of nanomaterials for their case study. This information should be brought to the next workshop for discussion.

Setting up wikis for each group

Students should provide the module tutor with their email addresses in this workshop to allow the module tutor to set up the wikis for each case study group. A guide to using a wiki is included in the appendix to this guide and depending on which wiki software you choose to use, instructions on how to set up the group workspaces will differ but they will be available using the help facility.

Workshop 4: Synthesis and cost of nanomaterials

Assignment to be completed during Workshop 4

Part 1: Introduction to using a wiki as a tool to support and assess the workshops

A computer lab is recommended for the first part of this which should take about one and a half hours. Some students may be encouraged to bring their laptops to class (particularly where wireless internet is available), but this is obviously at the discretion of both students and tutors concerned.

This workshop will explore the uses and versatility of wikis and sets out protocols for the wiki based project. All members of each group will have received invitations to their wiki by e-mail. Uploading files and editing page details can be practiced at this stage. Guidelines on using a wiki are provided in the appendix of both this guide and the student guide.

Most wikis allow conversion of the document to PDF form (Note: PDF formats do not allow for page numbers or word counts). In this manner, the students' wikis will form their final proposal and they will not be required to produce another separate document. It is important that during, or very soon after, this workshop each group set up their wiki appropriately, correctly identifying the page headings in line with the required content of their final proposal. They can then begin to populate it over the next number of workshops.

Examples of Nanotechnology wiki's

<http://tinyurl.com/nanochem24>

<http://tinyurl.com/nanochem25>

<http://tinyurl.com/nanochem26>

Part 2: Synthesis of nanomaterials

This part of the workshop will give students an overview of the synthesis of nanomaterials and help them to identify the instruments used to characterise the nanomaterials in their case study. It should take about one and a half hours. In this workshop the students should focus on the synthesis and costing of preparing their nanomaterial. Synthesis and/or characterisation of each nanomaterial is provided as an electronic resource. This should be used alongside their own research to identify a suitable method for the production of their nanomaterials.

Once a method has been agreed upon in the group, costing should be estimated based on preparation of 100 mL or 100 g of the selected nanomaterial. The costing can assume a yield of 90% for each synthetic step. They must remember to include VAT (20 %) on all raw materials to be purchased. ChemSpider provides a chemical vendors category for all compounds listed. Students should be reminded to consider whether a vendor will have to ship materials a long distance, the grade / specification for the chemical and the unit sizes that can be supplied. An excel spreadsheet similar to that shown below may be useful for this process.

Workshop 5: Introduction to Instrumentation

Assignment to be completed during Workshop 5

The aim of this workshop is for students to gain an overview of the type of instruments used to characterise a variety of nanomaterials. To achieve this, the students will be required to engage with the electronic resources provided, and update their wiki or PowerPoint presentation as a group to include three instruments suitable for characterisation of their assigned nanomaterial. If the wiki is used this should be prepared by the case study group as a whole and students may be graded on their individual input to the wiki.

Suggested instruments:

1. Atomic Force Microscope (AFM)
2. Scanning Electron Microscope (SEM)
3. Transmission Electron Microscope (TEM)
4. X-Ray Diffraction (XRD)
5. UV/Vis Spectrometer
6. Fluorimeter
7. Laser flash photolysis
8. Raman Spectrometer
9. Dynamic Light Scattering (DLS)

Images of each instrument and sample data have been supplied in the supporting electronic resource. The students will be requested to describe the instrument, comment on how it operates, what data it will provide, and the instrument limitations/sample considerations if any.

Student Learning objectives

- To provide an overview of an instrument capable of characterising nanomaterials.
- To create an information portal *via* a wiki/PowerPoint presentation with other members of their peer-group.
- To evaluate the importance of the data provided from the instrument.
- To construct a well designed instrument description from the information retrieved.
- To develop a time line for work.

Assignment to be completed before Workshop 6

Each case study group must write on three types of instruments from the list supplied that are suitable for analysis of their material and mention the following:

1. A description of the instrument along with an image.
2. Comments on how it operates.
3. What data it will provide.
4. The limitations of the instrument and sample requirements if any.
5. Time requirements for synthesis and analysis as a Gantt chart.

The details in Table 5 are provided to support students in production of a Gantt chart to plan the synthetic and analytical process. They should consider matters such as whether analysis is necessary during synthesis or whether it should be carried out after, if data is needed from one process before the next analytical step etc. An example of a Gantt chart is provided in the Appendix. It is desirable to carry out the work in the shortest possible timescale so learners should consider which tasks (if any) can be performed simultaneously. Multiple analysis runs may be needed for accurate results. Timings for synthesis steps should be deduced from experimental procedures.

Table 5: Timings for analytical procedures

Instrument	Time for analysis of 1 sample (min)
Atomic Force Microscope (AFM)	15
Scanning Electron Microscope (SEM)	15
Transmission Electron Microscope (TEM)	15
X-Ray Diffraction (XRD)	10
UV/Vis Spectrometer	5
Fluorimeter	5
Laser flash photolysis	5
Raman Spectrometer	5
Dynamic Light Scattering (DLS)	5

Workshop 6: Close of Module

In this workshop, each group will present their work, and will listen to and assess the presentations of the other groups. You may opt to invite some guest tutors to provide additional feedback. It is helpful to monitor the time and give each group a warning when they have one minute left.

It is important to emphasise to students that they have an essential role as an audience member for their peers. It is recommended that they are asked to provide a supportive environment by listening attentively, making some eye contact with the presenter, asking any questions they have in a respectful and non-confrontational way, and (if requested to) making some constructive comments on the peer feedback forms for the group assigned. These guidelines are based on the system used in the Toastmasters organisation, as are the feedback forms at the end of this section. Students should also be reminded that listening to the other group's presentations provides a very useful opportunity to see how others approached the same project, and to assess what they might incorporate from their methods in the future. Students should be prepared to receive constructive feedback from their peers and tutor, and be ready to take some notes on any corrections or recommendations.

It is at your discretion to decide whether to give each group some feedback in front of the entire group after they have presented or to give general feedback at the end.

The individual folders of work should be submitted in this workshop as evidence of the individual work done. You can use this when assessing individual contributions to group activities. Students should be reminded about the deadline for the submission of their reflective piece which will usually be several days after the presentation. Guidelines for the reflective piece are given in the student guide appendices.

The student should receive an individual form in which they are given feedback on each workshop assignment based on their folder of work (wiki) which is given an overall mark as shown in Table 6. A sample peer assessment form is provided in Table 7, which you may use during the class presentation of the wikis (The form is presented in duplicate so they can be easily printed two to a page.).

Table 6: Overall module assessment form and feedback.

Assignment	Assignment weighting	Mark	Strengths	Room for improvement
Information retrieval (individual)	10%			
Powerpoint presentation (group)	20%			
Debate (individual)	20%			
Case Study (group)	50%			

Table 7: Peer assessment form for wiki presentations

Case Study Group:				
Criteria (Tick box)	Very Good	Good	Average	Fair
Wiki layout				
Content & relevance				
Clarity & accuracy of content				
Visual impact				
References				
Comment on what they did well:				
Comment on where they could improve:				
Mark out of 10				

Case Study Group:				
Criteria (Tick box)	Very Good	Good	Average	Fair
Wiki layout				
Content & relevance				
Clarity & accuracy of content				
Visual impact				
References				
Comment on what they did well:				
Comment on where they could improve:				
Mark out of 10				

Appendices

Appendix 1: Wiki guidelines

A wiki is “a collaborative website consisting of one or more pages that allow authorized users to contribute to or edit page content.” (source: <http://tinyurl.com/nanochem29>)

Why use a wiki?

Wiki software is very easy to use, and allows students to work and to write collaboratively to produce a report/presentation/webpage. The wiki is a means of generating a very useful archive of all of the information that is relevant to the assignment as the project proceeds. It provides the added flexibility of being able to work anywhere where a PC or laptop and internet connection are available at any time.

All previous versions of each page can be accessed using the “Page History” function which means that no work can be permanently overwritten or deleted. Contributions made by each member can be easily tracked to assess their quality, quantity, and whether they were made across the entire timeframe of the assignment. Peer feedback and review is facilitated by the comments and page editing option. The assignments and the feedback provided can be accessed easily, and stored indefinitely for future reference and are available in a flexible format (pages can usually be saved as PDF’s). Wikis are regularly used in organisations to allow groups to collaborate on projects and documents, and to share knowledge and the ability to use one is a valuable transferable skill (see the references at the end of this section).

Available software

The authors have experience of using PBworks, but there are several other products and most virtual learning environments now have a built-in wiki. Should you wish to research options www.wikimatrix.org allows the user to select the wiki platform which best suits their needs in terms of a number of features, including access control and security, advertising, file sharing, formatting etc. PBworks has a basic version that is free to use (see <http://tinyurl.com/nanochem30> to sign up and <http://tinyurl.com/nanochem31> for help).

What do academic staff need to be able to do?

In advance:

The common tasks tutors perform initially are creating new wikis, adding group members to them and adding a message to the front page. You may decide to add some of the wiki pages the group will need by adding new pages and naming them appropriately (e.g. Introduction, Results, Discussion, Bibliography, Administration and Planning, Resources). However, it is less time consuming if the students are provided with a list of pages that they should add themselves instead. It may also be helpful to create one central wiki that all groups have access to where assignment details and general feedback can be posted, and all technical/academic queries can be dealt with.

This will avoid duplication. Video files (e.g. tutorials on software) and audio files can also be added if you wish to do so.

On an ongoing basis :

You may have to deal with some technical queries. It is recommended that students are required to confirm that they already asked a peer about their technical problem, and that they checked any guidelines they already have before they post a query to the tutor. Posting the answer in a central location accessible to all students saves considerable time, and builds up a “Frequently Asked Questions” page.

You should aim to provide some feedback on progress each week if possible. This would usually be posted as a comment on each group’s page, although general feedback on a central wiki can also be used if similar issues are cropping up or if time is an issue. **The first week is particularly important** as students may be reticent about being the first to write on a page and often need encouragement. Students may find it useful to add files with their draft work in advance of their weekly group meeting, and then decide what will be added to the page at their meeting. A selection of tutor comments from previous wiki assignments have been added at the end of this section. You may want to cut and paste some of these and/or begin to save some of your own to a Microsoft Word file to make this process easier.

What do students need to be able to do?

The common tasks students will perform are adding and editing wiki pages, adding comments and links to pages and inserting tables and chemical schemes/structures. Note that most chemical drawing software allows for structures to be saved as images and the format required by the wiki for embedding an image directly will be specified in the help menu (for PBworks wikis, images need to be in a PNG, JPEG, or GIF format). If students have not used wikis before, it is recommended that time be booked in a computer lab to allow them to practice these tasks on the wiki you have set up for each group.

This should only take 30 minutes maximum and requires you to have obtained student e-mail addresses in advance to set up the wikis.

Some issues with wikis

Netiquette and group interaction:

The concept of “netiquette” is discussed in the student guide, and is important to highlight at the outset, because of the lack of visual cues when not communicating face-to-face. Students should be respectful to each other and be conscious of not offending or insulting anyone. You may want to ask the students to suggest some ground rules about working in their groups, such as, remaining respectful towards a group member who is not contributing, providing constructive feedback to peers (e.g. posting a comment first before making changes to someone else’s work), and consulting with the group in relation to important decisions.

Plagiarism

Students may need to be reminded of the importance of providing references for information (and acknowledging the source of images), and of the need to use their own words to incorporate the ideas and information from the sources used into their report/presentation. Students can be asked to sign a declaration

such as the one below (see also “A Handbook for Deterring Plagiarism in Higher Education”, Jude Carroll, Oxford Centre for Staff and Learning Development, Oxford, 2002).

Alternatively, they can be asked to convert their wiki pages to pdf files and submit them to plagiarism detection software.

Example of a statement of originality;

We hereby affirm that

1. the research and writing of this report/presentation is entirely our work;
2. we have not intentionally plagiarised any portion of the report/presentation and have included quotation marks or references where required:

Signature: _____ Date: _____

Signature: _____ Date: _____

Signature: _____ Date: _____

Signature: _____ Date: _____

Suggested assessment criteria

Peer assessment criteria may be based on the frequency and quality of contributions. For tutors, assessment criteria that can be used for individual students can be based on effort and collaboration (see this video of two staff from Swinburne University of Technology discussing using wikis in education for more details <http://tinyurl.com/nanochem32>).

Suggested aspects to consider for wiki assessment are:

- Contribution to Group (based on wiki, summaries of meetings and workshop participation)*
- Effort (based on wiki, summaries of meetings and workshop participation)
- Peer Assessment (frequency and quality of contributions, both online and face-to-face)

* A detailed rubric for assessing this component is provided in the following reference which may be useful to consult: Learning to Teach Online, Case Study. Using wikis for student collaboration, Simon McIntyre, accessed 12 January 2012 at <http://tinyurl.com/nanochem33>

Further Reading/Viewing:

A video showing how to set up a PBworks wiki for educational use: <http://tinyurl.com/nanochem34>

References on use of wikis in organisations:

- “Corporate Wiki Users: Results of a Survey”, A. Majchrzak, C. Wagner & D. Yates, *WikiSym'06, Proceedings of the 2006 international symposium on Wikis*, D. Riehle, J. Noble, Eds. (ACM Press, 2006), vol. Odense, De, pp. 99-104, accessed 18 October 2011 at <http://tinyurl.com/nanochem35>
- A wiki to develop policy in the area of green chemistry in California is available here: <http://tinyurl.com/nanochem36> and here: <http://tinyurl.com/nanochem37> and <http://tinyurl.com/nanochem38>
- Ganfyd wiki is a medical database that can be edited by registered medical practitioners and viewed by anyone: <http://tinyurl.com/nanochem39>
- Drug Discovery Today article on use of an in-house wiki by Hoffman La Roche to share medicinal chemistry knowledge: Mayweg, A., Hofer, U., Schnider, P., Agnetti, F., Galley, G., Mattei, P., Lucas, M., Boehm, H. J.; ROCK: the Roche medicinal chemistry knowledge application - design, use and impact. *Drug Discovery Today* **2011**, 16 (15-16), 691-696.

Use of wikis in teaching and learning chemistry

- Paper discussing use of wikis to support PBL in chemistry: Williams, D. P., Woodward, J. R., Symons, S. L., Davies, D. L.; A Tiny Adventure: the introduction of problem based learning in an undergraduate chemistry course. *Chemistry Education Research and Practice* **2010**, 11 (1), 33-42.
- Article on using wikis to promote collaboration on online lab reports: Elliott, E. W., Fraiman, A.; Using Chem-Wiki To Increase Student Collaboration through Online Lab Reporting. *Journal of Chemical Education* **2010**, 87 (1), 54-56.
- Evans, M. J.; Moore, J. S., A Collaborative, Wiki-Based Organic Chemistry Project Incorporating Free Chemistry Software on the Web. *Journal of Chemical Education* **2011**, 88 (6), 764-768.

Some useful assessment and evaluation guidelines, learning factors and project design information:

- Tsai, W. T.; Li, W.; Elston, J.; Chen, Y. N., Collaborative Learning Using Wiki Web Sites for Computer Science Undergraduate Education: A Case Study. *Transactions on Education* **2011**, 54 (1), 114-124.

Sample Front page of Wiki (input from tutor needed where highlighted)

Welcome to your Nanochemistry Group Assignment Wiki ([Link to Index page](#))

Dear Group X,

Welcome to your wiki. You can begin to add relevant links and files on background material and your group meetings, and draft your [report/presentation](#) for your context/problem based learning assignment. Please take advantage of the **Help** link above to the right, and the links provided below by the software providers to ensure that you are using the wiki effectively. There are also some videos available on YouTube that show you how to carry out particular tasks.

Each group member can edit any wiki page or add a comment. The **Page History** link allows you to see previous versions of each page and recall portions of it if you want to do so. It also provides a permanent record of which group member did what. The **Pages & Files** tab on the top left has a number of existing templates that you can edit that you may find useful (e.g. meeting agenda). You can also create new pages with the formatting of your choice. Please use folders, link related pages, and name files and pages in a logical and structured way so that you can find information here easily. To help with this, your group are provided with names that should be used for the pages that will make up the main body of your [report/presentation](#). **There is a space limit on each wiki page. If you find that a page is no longer accepting edits, you have probably reached this limit. You will need to add a new page, and link to this new page from the end of the existing one to continue that section.**

You should receive daily email notifications of changes made to each page, but you can change that to a different interval if you wish to do so by altering the "Notification Preferences" settings at the bottom of your log-in page.

Please make sure that all members of your group have access to this wiki. If there is a problem, ask the person who has not received an invitation to the wiki to e-mail me at [tutorname@xxx](#) to request access.

Also, remember that this wiki is for academic use only, that all changes are saved and traceable, and all entries made by a student are used to determine the grade obtained.

I'm looking forward to seeing the work that your group produce,

[Tutor name.](#)

Welcome to PBworks

Need Help? We're here for you:

- [The PBworks Manual](#) can help show you how to edit, add videos and invite users.
- The best way to get your support questions answered is to **click the help link** at the top of this page. Our support gurus will get back to you asap.

Sample feedback comments from wikis used previously

Initial comments

- This is a reminder to start to use this wiki to plan your group assignment. Please make sure that you discuss it as a group (face to face or in an online chat) and assign tasks. Good luck with your assignment.
- You've made a good start here. Hopefully the other people in your group will also begin to contribute soon.
- This looks good so far. Can you add the reference numbers in below the relevant images though, and add them into the text where relevant too.
- Your page is coming along very well. You've done a lot of research so far and found some interesting information.

Positive comments

- In general, the pages are well researched and referenced.
- Relevant schemes and images have been used, and they have been referred to well.
- Visually, the pages made an impact.
- Well done on preparing a well researched and clearly structured report/presentation with relevant schemes and images that all team members contributed too.
- Your work has been carefully checked and proofread, and the wiki page history shows that you were all contributing.

Room for improvement

As far as possible, any comment on an improvement that could be made was prefaced by a positive comment on another aspect of the wiki.

- There is some repetition of information - particularly about xxxxx.
- The overall report/presentation is quite long and is in need of a final edit - for structure and content and language errors.
- It is important that a group make sure they have time to review their work together towards the end, edit it to make sure that there is no repetition, and that all information can be clearly understood.
- Only include information that you understand yourself and explain all scientific and technical terms which would be unfamiliar to your peers.
- It would have been nice to see more textbook references instead of websites.
- Where appropriate, add references for information you have given.
- Be careful that you have put all information in your own words and haven't plagiarised.

- A diagram/table/scheme in the section on xxxxxx would be helpful.
- Information on xxxxx was not included. Please add this before submission of the final version. Include a few more chemical structures.
- Look over where xxxxx is discussed. It could be made clearer.
- Make sure that one or two people edit the whole report/presentation at the end so there is a "flow" between the sections and a similar style is used all the way through.
- Give the information required in the correct format in the references e.g. article authors/journal name/place published for a text book.

General Feedback and Technical Support Comments

Please post any queries about any technical problems or general help you need on this page.

- Most of you made a good attempt at xxx. The aspects that were often not included were: ...
- These requirements were mentioned on page xxxxx of your assignment guidelines and should be included in the final report.
- Give the references you used for the information about xxxxx.

Suggested layout of Wiki

The screenshot shows a PBworks Wiki page titled "NanoChemistry Group Assignment Wiki". The page content includes:

- A "Welcome" message from the tutor, including instructions on how to edit and add content.
- A "Dear Group" message with specific instructions and a space limit warning.
- A "Please make sure" instruction regarding access and academic use.
- A "Welcome to PBworks" section with a "Need Help?" link and a list of resources.
- A "Recent Activity" sidebar showing a log of recent edits: "Home" edited by Michael, "FrontPage" renamed by Michael, and "SideBar" edited by Michael.

Annotations on the image:

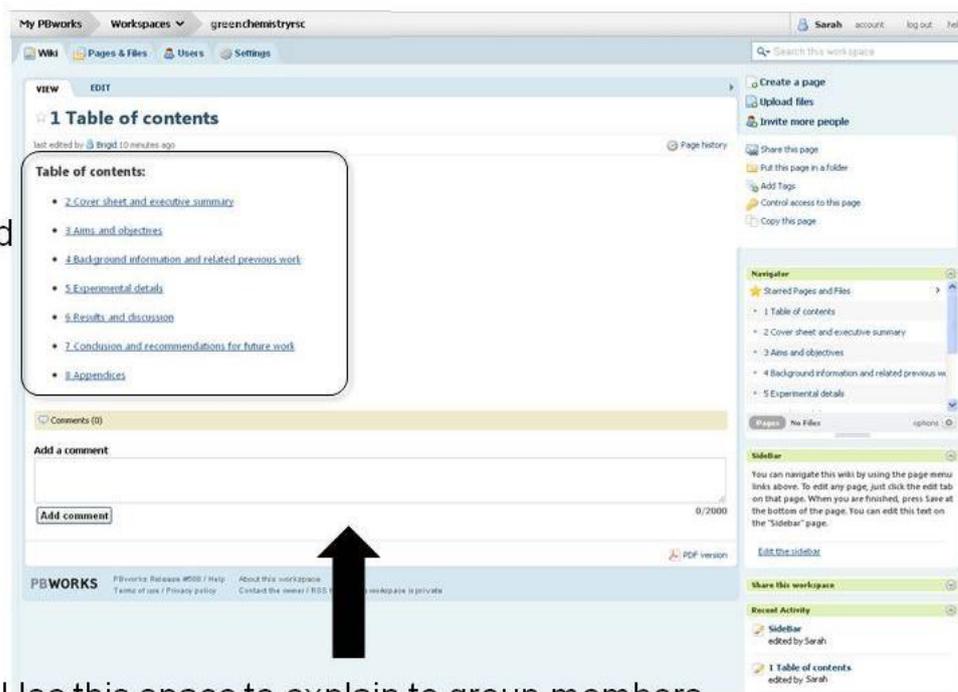
- An arrow points to the introductory text from the tutor.
- An arrow points to the "Recent Activity" sidebar, labeled as a log of recent activity used to monitor student contributions.
- An arrow points to the "SideBar" navigation menu, labeled as "Pages created using project headings".

Introductory text
from tutor

Log of recent activity used to
monitor student contributions

Pages created
using project
headings

Pages can be edited and linked to other pages throughout the wiki.



Use this space to explain to group members reasoning for changes made

Appendix 2: Sample Gantt chart

TASK	Day 1 -4	Day 5 - 8	Day 9 – 13	Day 13 – 17	Day 18 - 22	Day 22 - 25
Synthesis of nanomaterial	█					
Characterisation step 1		█				
Characterisation step 2		█	█			
Characterisation step 3				█	█	

Appendix 3: Student wiki/presentation checklist

The following are the names of the pages that should be used to make up the main body of the wiki/presentation:

- Project overview (300 words)
 - Table of contents (with links to the other pages) or presentation overview
 - Aims and objectives of project
 - Brief background on case study
 - Application of selected nanomaterial
- Method and cost of nanomaterial preparation (1500 words)
- Characterisation of the nanomaterial (1000 words)
 - What analysis will be done
 - Information it will provide
 - Constraints of method
- Gantt chart
- Conclusion (200 words)

There are also a number of pages that each group should consider adding to the wiki that will help with planning and communication. These are:

- Project planning and meetings - Use this page to record any tasks agreed within the group.
- Ideas and suggestions - Use this page as a sounding board and suggestion box for general issues.
- Annotated bibliography - Use this page to post links or citations to useful websites, videos, articles or textbooks. Each one must include a short summary of why this information source is useful and refer to a specific page/section in it if necessary.

Appendix 4: Guidelines for student reflective piece

In this short report (500 to 800 words approx), students should:

1. Briefly describe their role in the case study project and the contribution they made.
2. Discuss how they experienced working in a team (considering both the positive and negative aspects).
3. Discuss any changes that they would make to how they and their group went about the case study project if they were repeating it.
4. Summarise what they found to be the most interesting aspect of the entire project as well as the most challenging aspect (presentation, debates, group work etc.).
5. Consider whether they think the project was useful to their learning and whether all of the learning outcomes were met.
6. Assess whether they have developed the transferable skills listed at the beginning of the student guide further as a result of this project. Highlight any that they think are particularly important or that they have now gained confidence in.
7. Consider whether they have found that writing a reflective piece like this helps them to review what they learned over the course of the project.

This reflective piece is assessed based on;

- **content** (60% - there are no right or wrong opinions but they must make sure that they discuss **all** of the topics listed above)
- **presentation** (10%) and
- **coherence, accuracy and structure** (30%).

Appendix 5: Case Studies

The following is provided for each case study:

- Overview including context and report template
- Annotated bibliography (only in tutor edition)
- Nanomaterial synthesis and/or characterisation summary (included in tutor guide and available to students as download from relevant electronic resource)

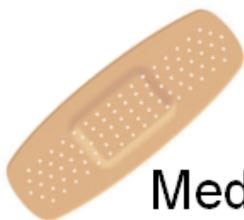
The bibliography provided in this tutor guide can be given to students at your discretion if you feel they are struggling in the information retrieval tasks.

Detailed synthetic procedures for synthesis of the selected nanomaterials are given in Appendix 5, including materials, safety information, methods and technical notes. Again, these can be provided to students at your discretion, or can be applied in laboratory sessions should you wish to alter the resource.

Case studies:

1. Mediwraps - Silver nanoparticles in wound dressings
2. Goldglow - Gold nanoparticles as probes to detect cancer biomarkers
3. Kleeenview - Titanium dioxide in self cleaning windows
4. Nanoklenz - Carbon Nanomaterials in water filters
5. Solarsunrayz - Quantum Dots for solar energy capture in solar panels

Case study 1: Mediwraps



® Metalopharm

Mediwraps: The future of wound dressing

Metalopharm is a pharmaceutical company which is currently investigating two applications of metal nanoparticles. Project Mediwraps uses silver nanoparticles to form antimicrobial coatings on their plasters/bandages. Metalopharm wish to investigate the viability of silver nanoparticles in wound dressing, and to learn more about the preparation, characterisation and costing of this material.

Students are required to prepare a group wiki that deals with the content specified in a project description template as shown below.

Project Description Template

Project overview [Abstract/Summary] (max 300 words) Aim of the project. Overview of nanomaterial, including relevant background information.
Product Preparation (max 1500 words, may include reaction schemes and diagrams) Protocol for the preparation of the product (this should include any interesting observations/properties of the product).
Product Analysis (max 1000 words) What analysis will be done? What information this will provide?
Timelines Timelines of analysis to be carried out to include report writing (present in a Gantt chart format).
Costing How much will the raw materials cost.
Conclusion (max 200 words) Summarise results and recommendations

Silver nanoparticles annotated bibliography

Nanotechnology and human health: risks and benefits; Cattaneo, A.G., Gornati, R., Sabbioni, E., Chiriva-Internati, M., Cobos, E., Jenkins, M.R., Bernardini, G.; *Journal of Applied Toxicology*, 2010, 30, 730-744

While regulatory agencies are very careful when approving new nanomaterials for human use because of their potential toxicity, concerns are rising that the widespread research on new nanomaterials that are potentially useful in medicine has not been counterbalanced by an adequate knowledge of their pharmacokinetics and toxicity. This article discusses nanomaterials used as carriers in medicine and the construction of databases of reliable toxicity information, with a glimpse of potential nanomedicines currently being researched.

At the nanoscale: nanohemostat, a new class of hemostatic agent; Ellis-Behnke, R.; *Wiley Interdisciplinary Reviews-Nanomedicine and Nanobiotechnology*, 2011, 3, 70-78

This paper discusses the use of nanomaterials in medicine, in particular as hemostatic agents to stop bleeding in tissue. Several nanomaterials have been reported to be useful in drug delivery and encapsulating therapeutic agents, but their use as hemostatic agents overcomes many issues related with current treatments such as chemical agents which promote clotting, thermal devices and mechanical methods. This review focuses on the 'nanohemostat', a new class of agent that stop bleeding in less than 15 seconds. By using a synthetic biological material that self-assembles at the nanoscale when applied to a wound, a nanofibre barrier is formed to stop bleeding in any wet ionic environment in the body. Furthermore, the material is broken down into natural l-amino acids that can be used by the surrounding tissue for repair or excreted in urine. This new technology may be able to significantly reduce the time spent in surgery stemming bleeding and hence revolutionise the field.

Nanosilver: A nanoparticle in medical application; Chen, X., Schluesener, H.J.; *Toxicology Letters*, 2008, 176, 1-12

Nanosilver is one of only a few nanoparticle products which are currently in use for medical purposes. Due to its strong antibacterial activity, silver nanoparticles (AgNPs) are being incorporated on to various textiles. Further, nanosilver is used for treatment of wounds and burns or as a contraceptive and marketed as a water disinfectant and room spray. This review focuses on major questions associated with the increased medical use of nanosilver and related nanomaterials, such as the interaction of AgNPs with the human body after entering, biodistribution, organ accumulation, degradation and possible adverse effects.

Does the Antibacterial Activity of Silver Nanoparticles Depend on the Shape of the Nanoparticle? A Study of the Gram-Negative Bacterium *Escherichia coli*; Pal, S., Tak, Y.K., Song, J.M.; *Applied and Environmental Microbiology*, 2007, 73, 1712-1720

While other publications have linked the size of AgNPs with their effectiveness against bacteria, this paper investigates the shape dependence of the antibacterial activity against *Escherichia coli*. Of the shapes studied, truncated triangular silver nanoplates displayed the strongest biocidal action, compared with spherical and rod-shaped nanoparticles and with Ag⁺ (in the form of AgNO₃).

An evidence-based environmental perspective of manufactured silver nanoparticle in syntheses and applications: A systematic review and critical appraisal of peer-reviewed scientific papers; Tolaymat, T. M., El Badawy, A. M., Genaidy, A., Scheckel, K. G., Luxton, T. P., Suidan, M.; *Science of the Total Environment*, 2010, 408, 999-1006

This review covers concerns in the regulatory and research communities about potential environmental impacts associated with the use of silver nanoparticles. The primary objective of the study is to present an evidence-based environmental perspective of silver nanoparticle properties in syntheses and applications. Environmental perspectives examined include the salt precursors and agents used in synthesis of AgNPs, the characteristics of AgNPs when integrated into nanocomposites and bimetallic nanoparticles, and the morphology of AgNPs.

Non aggregated colloidal silver nanoparticles for surface enhanced resonance Raman spectroscopy; Power, A.C., Betts, A.J., Cassidy, J.F.; *Analyst*, 2011, 136, 2794-2801

Silver nanoparticles with a tuneable λ max were produced as colloids by heterogeneous nucleation. The synthesis process is both fast and repeatable, and suitable for application in an undergraduate teaching environment.

Silver nanoparticles summary report (including preparation and analysis)

Potential application:

Anti-bacterial agents to be incorporated into hygienic surfaces, wound treatments and medicines.

Preparation method:

Seed production:

1. 2 cm^3 of 0.001 M AgNO_3 was added to 2 cm^3 of poly vinyl alcohol (PVA) (1% w/v) and mixed well.
2. While stirring constantly, 2 cm^3 of 0.001 M NaBH_4 was added drop wise to this solution.
3. The resulting solution was a golden yellow.

Fabrication of coloured colloids:

1. 1 cm^3 of the seed solution, 3 cm^3 0.1 M tri-sodium citrate (TSC) and 5 cm^3 $0.1\text{ M N}_2\text{H}_4$ was added to 1 cm^3 PVA (1% w/v) making sure to stir well.
2. Coloured colloids were produced by adding 0.001 M AgNO_3 while continuing stirring of this 'growth mixture'.

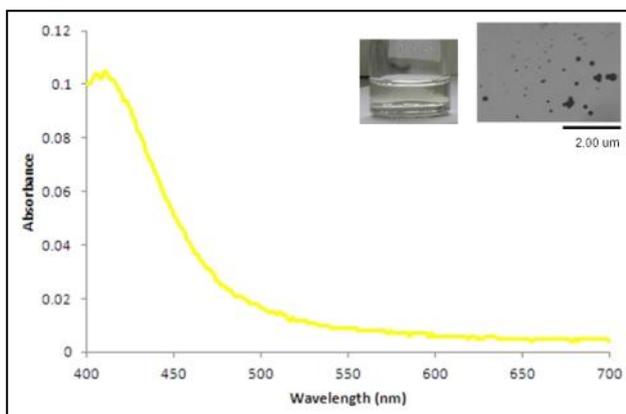
Results:

Six colloid solutions were prepared: yellow, orange, red, purple, blue and green.

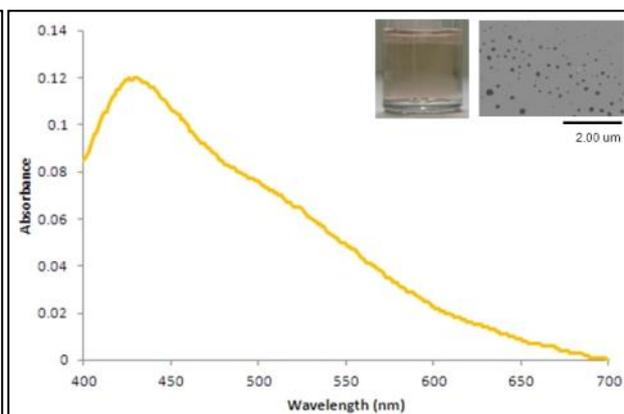


Silver nanoparticle colloid solutions

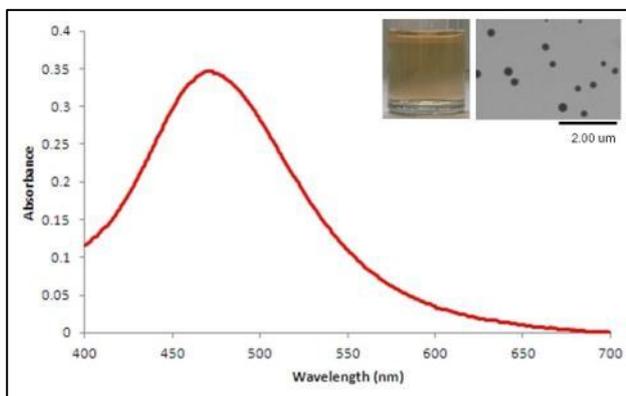
UV-Vis spectroscopic analysis was carried out on each solution, and dynamic light scattering and transmission electron microscopy were also performed to determine size of particles in the colloid solution.



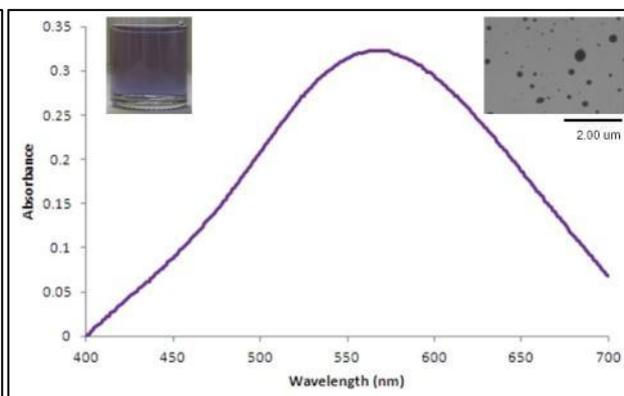
Analytical results for yellow colloid



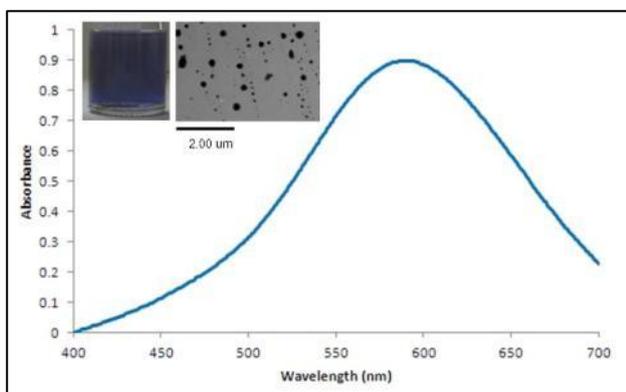
Analytical results for orange colloid



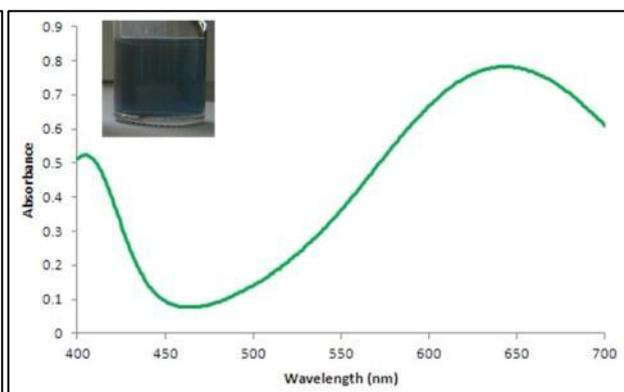
Analytical results for red colloid



Analytical results for yellow colloid

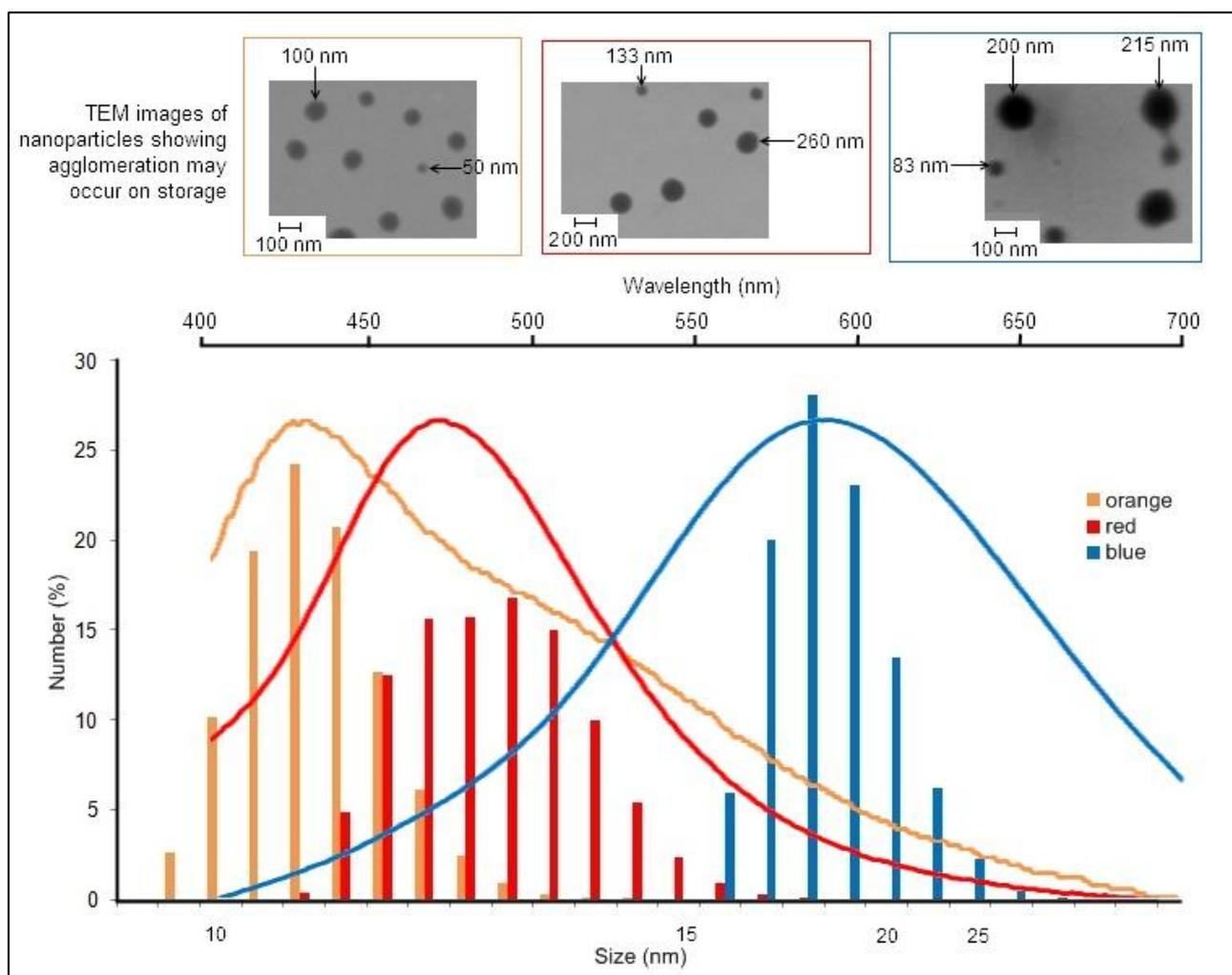


Analytical results for blue colloid



Analytical results for green colloid

It is clear that agglomeration has occurred within the colloid solutions upon storage as the particle sizes are larger than expected. Nonetheless, a clear trend can be seen correlating increase in size and the visible absorbance maximum. Therefore, visible spectroscopy can be used as a simple indication of relative particle size distribution in solutions. For example, the green solution contains a mixture of yellow and green colloid solutions as can be seen from the visible spectrum, so it can be predicted that it contains a mixture of both these small and large nanoparticles. This theory is supported by comparison with results from dynamic light scattering sizing of particles. As can be seen below, shifts in sample wavelengths correlate well with changes in particle size.



Relationship between colloid colour and size of nanoparticles

Case study 2: Goldglow

Metalopharm



Goldglow: Pioneers in cancer diagnosis

Metalopharm is a pharmaceutical company which is currently investigating two applications of metal nanoparticles. Project Goldglow involves using gold nanoparticles as ultrasensitive fluorescent probes to detect cancer biomarkers in human blood. The project brief is to investigate the viability of gold nanoparticles in biomedical applications, and learn more about the preparation, characterisation and costing of this material.

Students are required to prepare a group wiki that deals with content specified in project description template as shown below.

Project Description Template

Project overview [Abstract/Summary] (max 300 words) Aim of the project. Overview of nanomaterial, including relevant background information.
Product Preparation (max 1500 words, may include reaction schemes and diagrams) Protocol for the preparation of the product (this should include any interesting observations/properties of the product).
Product Analysis (max 1000 words) What analysis will be done? What information this will provide?
Timelines Timelines of analysis to be carried out to include report writing (present in a Gantt chart format).
Costing How much will the raw materials cost.
Conclusion (max 200 words) Summarise results and recommendations

Gold nanoparticles annotated bibliography

Nanotechnology and human health: risks and benefits; Cattaneo, A.G., Gornati, R., Sabbioni, E., Chiriva-Internati, M., Cobos, E., Jenkins, M.R., Bernardini, G.; *Journal of Applied Toxicology*, 2010, 30, 730-744

While regulatory agencies are very careful when approving new nanomaterials for human use because of their potential toxicity, concerns are rising that the widespread research on new nanomaterials that are potentially useful in medicine has not been counterbalanced by an adequate knowledge of their pharmacokinetics and toxicity. This article discusses nanomaterials used as carriers in medicine and the construction of databases of reliable toxicity information, with a glimpse of potential nanomedicines currently being researched.

Gold nanoparticle-based signal amplification for biosensing; Cao, X. D., Ye, Y. K., Liu, S.; *Q.Analytical Biochemistry*, 2011, 417, 1-16

It is highly important that biological assays for monitoring processes such as protein interactions are highly sensitive. New methods of labelling are constantly being researched to enhance detection sensitivity, amongst which is the use of nanomaterials to increase the upload of electrochemical tags. Colloidal gold nanoparticles (AuNP) have been widely reported as labels for analytical signal amplification and biomedical use. These nanoparticles are produced under aqueous conditions by reduction of chloroauric acid (HAuCl₄). The resulting AuNPs have a number of advantages including rapid and facile synthesis, efficient surface modification by bioligands, narrow size range, and desirable biocompatibility. This paper provides an overview of recent advances in biosensing and biorecognition using AuNPs as labels, discussing signal amplification routes, and the main detection techniques and methods involved in their application.

Using the Enzymatic Growth of Nanoparticles to Create a Biosensor. An Undergraduate Quantitative Analysis Experiment; Bai, J., Flowers, K., Benegal, S., Calizo, M., Patel, V., Bishnoi, S. W.; *Journal of Chemical Education*, 2009, 86, 712-714

This paper describes a simple procedure for testing the biosensing potential of AuNPs. The experimental setup is divided into two parts in which the students first test a model chemical system and then simulate a glucose biosensor using the enzyme glucose oxidase. The reaction results in an increase in the size of the AuNPs. The optical extinction also increases due to a combination of the absorption and scattering from the particles in solution and is monitored by UV-Vis spectroscopy as a measure of the effectiveness of the sensing system. The experiments provide students with an opportunity to learn about the concept of biosensor design, and exposes them to the size dependent properties of AuNP extinction.

Gold nanoparticles summary report (including preparation and analysis)

Potential application:

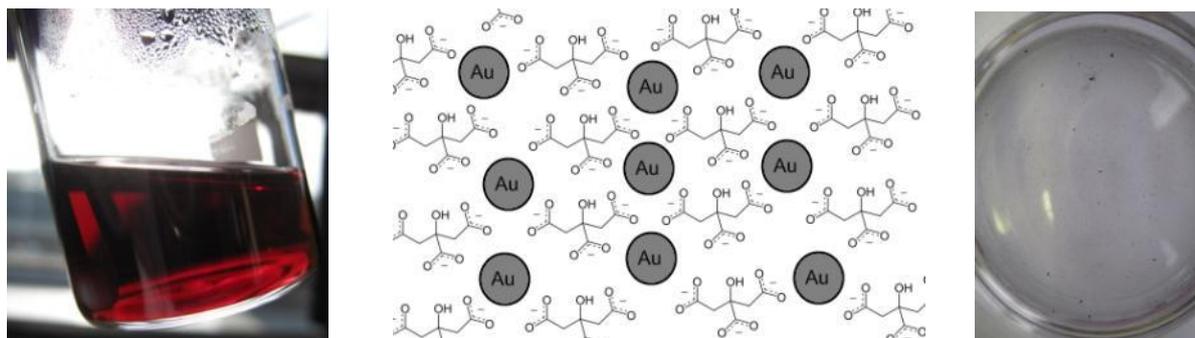
Biomarker for diagnostic medicinal use.

Preparation method:

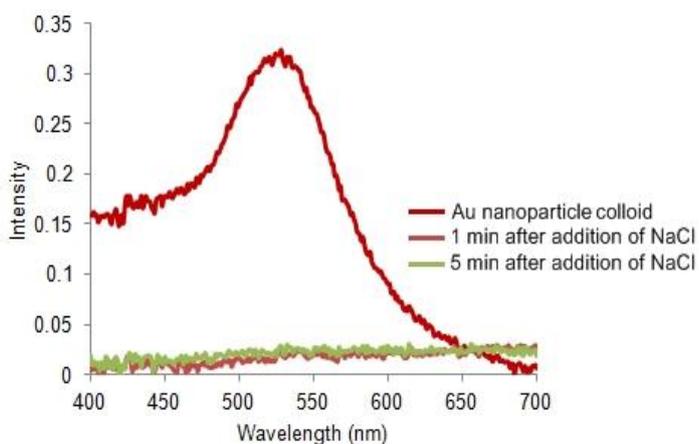
1. Prepare stock solutions of 1.0 mM HAuCl_4 and 38.8 mM $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7$ (tri-sodium citrate) in distilled water.
2. Heat 20 cm^3 of 1.0 mM HAuCl_4 to boiling while stirring with the magnetic stir bar.
3. As the solution begins to boil, add 2 cm^3 of 38.8 mM $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7$, continuing to boil and stir the solution until it is a deep red color (about 10 min). Add distilled water as needed to keep the total solution volume constant at 22 cm^3 .
4. When the solution is a deep red colour, turn off the hot plate and stirrer. Cool the solution to room temperature.
5. To test the effect of electrostatic forces, a few drops of 1 M NaCl may be added to the solution. Stir for approximately 10 minutes.

Results:

A red solution is formed containing the gold nanoparticle colloid. The colloid system is stabilised by citrate ions which prevent the gold particles from agglomerating and falling out of solution. Addition of an electrolyte such as NaCl can disrupt this system and destroy the colloid.



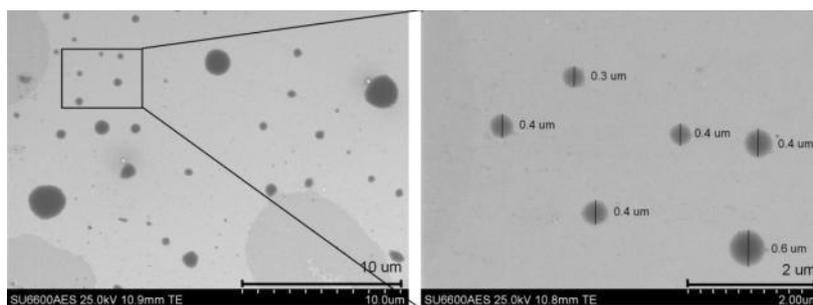
A) Gold nanoparticle colloid B) stabilising effect of citrate ions which prevent agglomeration C) aggregated particles which have fallen out of solution after addition of NaCl



Visible absorption of the colloid solution changes upon addition of the NaCl. This shows that within 1 minute there is already significant loss of the nanoparticles from solution.

Visible spectrum of gold colloid solutions

Transmission electron microscopy (TEM) was used to estimate the size of the gold nanoparticles. The large range of sizes suggests that the particles have begun to agglomerate, and may not be forming a stable colloid.



TEM images of the gold nanoparticles

Case study 3: Kleenview



Self-cleaning windows

Kleenview are a company that manufactures self cleaning windows. They are looking into the area of TiO_2 and photocatalysis, and wish to investigate the viability of TiO_2 as a photocatalyst and learn more about the preparation, characterisation and costing of this material.

Students are required to prepare a group wiki that deals with the content specifies in a project description template as shown below.

Project Description Template

Project overview [Abstract/Summary] (max 300 words) Aim of the project. Overview of nanomaterial, including relevant background information.
Product Preparation (max 1500 words, may include reaction schemes and diagrams) Protocol for the preparation of the product (this should include any interesting observations/properties of the product).
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Conclusion (max 200 words) Summarise results and recommendations

Titanium dioxide annotated bibliography

Nanotechnology for Site Remediation Fact Sheet (EPA 542-F-08-009); US Environmental Protection Agency <http://www.epa.gov/tio/download/remed/542-f-08-009.pdf> October 2008, last accessed 13.10.2011 (17 pages)

This document presents an overview of nanotechnology and its current uses in remediation. It presents information to help end users understand the potential applications of this group of technologies at their EPA sites. Topics covered include descriptions of nanoparticles used in site remediation, chemistry and applications of these nanoparticles, limitations on their use, toxicity and fate at end of life, cost, performance and examples of available products and existing users. This may be useful to provide context on this topic.

A review of water treatment membrane nanotechnologies; Pendergast, M.T.M., Hoek, E.M.V., *Energy Environ. Sci.*, 2011, 4, 1946-1971

This review reports uses of nanotechnology to enhance conventional water treatment membrane materials through various avenues. Examining enhancement potential and readiness for commercialisation, numerous technologies are ranked on a semi-quantitative scale, including zeolitic and catalytic nanoparticle coated ceramic membranes, hybrid inorganic–organic nanocomposite membranes, and bio-inspired membranes such as hybrid protein–polymer biomimetic membranes, aligned nanotube membranes, and isoporous block copolymer membranes. Performance enhancement criteria considered water permeability, solute selectivity, and operational robustness, while commercial readiness criteria examined material costs, scalability, and compatibility with existing manufacturing systems. Nanocomposite membranes were found to offer significant performance enhancements, with some products already commercially available. Key hurdles to commercialisation were identified for the various membrane nanotechnologies.

Environmental Applications of Nanotechnology and Nanomaterials; Power, T., Quiroga, B.; Presentation, last accessed 18.10.11 http://research.che.tamu.edu/groups/Seminario/materials/G10_Environmental.pdf

This presentation gives a broad introduction to applications of nanotechnology across the field of environmental science. Applications discussed include water remediation, fuel cells, solar panels, super capacitors, nuclear waste cleanup, growth promotion and many more.

Nano-adsorbents for the removal of metallic pollutants from water and wastewater; Sharma, Y.C., Srivastava, V., Singh, V.K., Kaul, S.N., Weng, C.H.; *Environmental Technology*, 2009, 30, 583-609

Recently there has been emphasis on the application of nanoparticles and nanostructured materials as efficient and viable alternatives to activated carbon as an adsorbent in waste water treatment. Titanium dioxide (TiO₂) nanoparticles are discussed in this article along with other nano-adsorbents, with special emphasis given to the removal of the metals Cr, Cd, Hg, Zn, As, and Cu. Separation of the used nanoparticles from aqueous solutions and the health aspects of the separated nanoparticles is also discussed.

Photocatalytic Applications of Micro- and Nano-TiO₂ in Environmental Engineering; Kwon, S., Fan, M., Cooper, A.T., Yang, H.; *Critical Reviews in Environmental Science and Technology*, 2008, 38, 197-226

The potential benefits of photocatalysis in the breakdown and removal of variety of environmental pollutants has been widely reported. TiO₂ has received particular attention for the removal of organic and inorganic compounds in the environment due its high photocatalytic activity, biological and chemical inertness, resistance to photocorrosion and low cost. Furthermore, TiO₂ photocatalysts exhibit high activity for the oxidation of volatile organic compounds (VOCs) under ultra violet light (UV), offering a technically practical and economic means to clean air and water. This review provides an overview of the principles governing environmental applications of micro and nano TiO₂ as a photocatalyst. The specific mechanisms and properties associated with nano TiO₂ are also discussed.

Ecotoxicity of nanosized TiO₂. Review of in vivo data; Menarda, A., Drobneb, D., Jemec, A.; *Environmental Pollution*, 2011, 159, 677-684

This report presents a review of data on the effect of nanoparticulate TiO₂ on algae, higher plants, aquatic and terrestrial invertebrates and freshwater fish. No discernable correlation was established between primary particle size and toxic effect. It is suggested that secondary particle size and particle surface area may be relevant to biological potential of nanoparticles, but insufficient confirmatory data exists for this theory to be confirmed. The report calls for better reporting of physicochemical characteristics of the nanoparticles in order to understand their biological reactivity and nanotoxicity. A number of practical measures are suggested which should support the generation of reliable quantitative structure–activity relationship (QSAR) models and so overcome this data inadequacy.

Synthesis of High-Temperature Stable Anatase TiO₂ Photocatalyst, Pillai, S.C., Periyat, P., George, R., McCormack, D.E., Seery, M.K., Hayden, H., Colreavy, J., Corr, D., Hinder, S.J.; *J. Phys. Chem. C*, 2007, 111, 1605-1611

This paper provides a method of production of nitrogen doped titanium dioxide to produce high levels of anatase material.

Titanium dioxide summary report (including preparation and analysis)

Potential application:

Photo-catalyst

Preparation method:

Titanium dioxide:

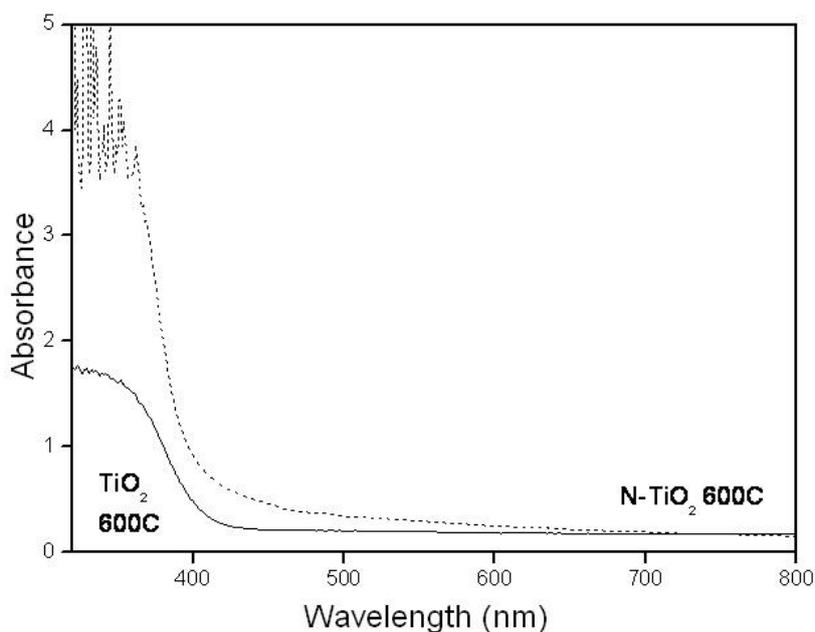
- Add 25 cm³ of 90 mM titanium isopropoxide to 48 cm³ of 0.8 M glacial acetic acid stirring on a magnetic stirrer plate.
- Add 150 cm³ of deionised water drop-wise to the partially hydrolysed titanium isopropoxide-acetic acid mixture. The solution will turn white. Ensure to maintain vigorous stirring.
- Stir the solution until a clear transparent solution or sol is formed; this may take up to 8 hours.
- Dry overnight at 100°C in an oven vented into a fumehood.
- Calcine the final product in air for 2 hours at 600 °C (anatase phase) and 700 °C (rutile phase) (ramp rate 5 °C min⁻¹).

Nitrogen doped titanium dioxide:

- Add 23.4 cm³ of 90 mM titanium isopropoxide to 206 cm³ of isopropanol stirring on a magnetic stirrer plate.
- Add 5 g of urea dissolved in 35 mL of distilled water. Ensure to maintain vigorous stirring.
- Stir the solution for 5 minutes.
- Dry overnight at 80 °C.
- Calcine the final product in air for 2 hours at 400 °C (ramp rate 5 °C min⁻¹).

Results:

The improved ability of N-doped TiO₂ to absorb visible light is shown in the absorbance spectrum, leading to increased photocatalytic activity over anatase TiO₂.



Absorbance spectrum of anatase TiO₂ and N-TiO₂

Case study 4: Nanoklenz

Nanoklenz

Cleaner water quicker



Nanoklenz are a company that manufactures water filters. They are looking into the area of the use of carbon nanomaterials in water filters for water remediation. Nanoklenz wish to investigate the viability of carbon nanomaterials in this application, and learn more about the preparation, characterisation and costing of this material.

Students are required to prepare a group wiki covering the content of a project description template as shown below.

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This review reports uses of nanotechnology to enhance conventional water treatment membrane materials through various avenues. Examining enhancement potential and readiness for commercialisation, numerous technologies are ranked on a semi-quantitative scale, including zeolitic and catalytic nanoparticle coated ceramic membranes, hybrid inorganic–organic nanocomposite membranes, and bio-inspired membranes such as hybrid protein–polymer biomimetic membranes, aligned nanotube membranes, and isoporous block copolymer membranes. Performance enhancement criteria considered water permeability, solute selectivity, and operational robustness, while commercial readiness criteria examined material costs, scalability, and compatibility with existing manufacturing systems. Nanocomposite membranes were found to offer significant performance enhancements, with some products already commercially available. Key hurdles to commercialisation were identified for the various membrane nanotechnologies.

Nanotechnology in Groundwater Remediation; Rajan, C.S.; *International Journal of Environmental Science and Development*, 2011, 2011, 2, 182-187

This review gives a perspective on the use of nanoparticles to solve water treatment issues. It cites a number of potential benefits nanotechnology holds over conventional treatment such as shorter treatment times, suitability for use *in-situ*, and improved effectiveness. Topics discussed include various aspects of research and experimental works in this area using nanoparticles such as Zero valent iron (nZVI) and Carbon Nanotubes (CNT). It also looks at the potential deleterious effects of using these nanoparticles.

Carbon nanotube sponge filters for trapping nanoparticles and dye molecules from water; Li, H., Gui, X., Zhang, L., Wang, S., Ji, C., Wei, J., Wang, K., Zhu, H., Wu, D., Cao, A.; *Chem. Commun.*, 2010, 46, 7966-7968

This short communication reports the use of porous sponge like materials composed of a 3D carbon nanotube network to filter nanoparticles and dye molecules from water. The interconnected structure can trap molecules by physisorption without further chemical functionalisation offering potential materials for the remediation of contaminated water.

Dai, L., Carbon Nanotechnology - Recent Developments in Chemistry, Physics, Materials Science and Device Applications. 2006, Elsevier.

This introductory text covers the development of carbon nanotubes and buckminster fullerene as important nanomaterials. Chapter 1 gives a broad overview, with an entire section covering the preparation of these materials, and Chapter 14 covering the use of carbon nanomaterials in filtration systems.

Allotropes of carbon as nanomaterials summary report

Historically carbon is classified into three principal forms (allotropes):

- diamond
- graphite
- amorphous carbon

The properties of these materials vary depending on the arrangement of the carbon atoms.

Principle allotropes of carbon

Allotrope	Structure	Properties
Diamond	3D network of C atoms, each bonded to 4 others	Hard, clear
Graphite (pencil "lead")	Stacked layers of C in honeycomb lattices	Soft, black
Amorphous carbon	No crystal structure	Black powder

There has been great progress in the understanding of carbon structures since a single sheet of graphite, known as graphene, was first described in 1962. The era of carbon nanotechnology began in 1985 with the discovery of buckminsterfullerene by Kroto *et al*, followed by the production of carbon nanotubes by Iijima *et al* in 1991. Figure 1 shows an overview of the structures of the carbon allotropes.

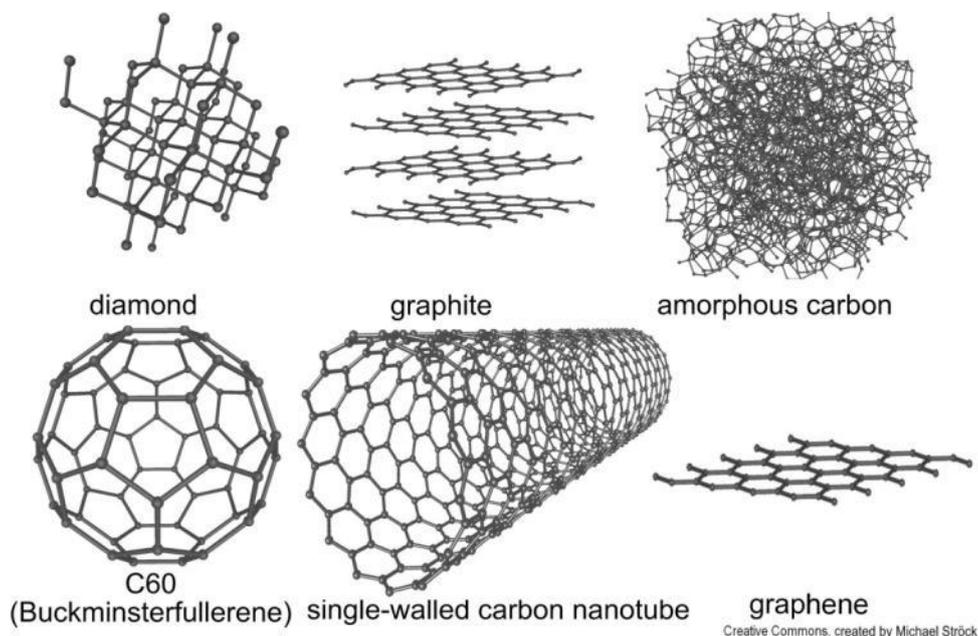


Figure 1: Carbon allotropes

Summary of carbon allotrope nanomaterials

Graphene

- Flat single layer of carbon atoms in a honeycomb lattice
- Stacks to form graphite
- Rolled to form carbon nanotubes
- Wrapped up to form buckyballs

Properties:

- good thermal and electric conductor
- incredibly strong

Applications:

- semiconductor circuits and computer parts (transistors, electrodes, ultra-capacitors)
- single-molecule gas detection
- nanoribbons
- solar cells
- biodevices
- anti-bacterial materials

Carbon nanotubes (CNTs)

- Single or multiple graphite sheets rolled up into nanoscale tube
- Single-wall carbon nanotubes – SWNTs
- Multi-wall carbon nanotube, MWNTs
- Semiconducting or metallic behaviour depending on:
 - arrangement of the graphite rings
 - diameter of the tube
 - degree of twists of the graphene sheet
- Unique electronic, magnetic, mechanical, and optical properties

Applications:

- novel single-molecular transistors
- molecular computing elements
- electron field emitting flat panel displays
- gas and electrochemical storage
- molecular-filtration membranes
- artificial muscles

Buckminsterfullerene (C₆₀) – Bucky balls

- Discovered in 1985 by Kroto *et al* (Nobel Prize 1996)
- Carbon molecule with a soccer-ball-like structure
- 12 pentagons and 20 hexagons facing symmetrically
- Can modify surface or hollow centre

Applications:

- superconductors
- hydrogen storage
- high-efficiency solar cells
- chemical sensors

Buckypaper

- Thin sheet of aggregated carbon nanotubes
- Inadvertently developed in Richard Smalley's lab
- Nanotubes in liquid suspension are filtered through a fine mesh
- The nanotubes stick together forming a thin film disk

Properties:

- “harder than diamonds”
- “stronger than steel at a fraction of the weight”

Potential applications:

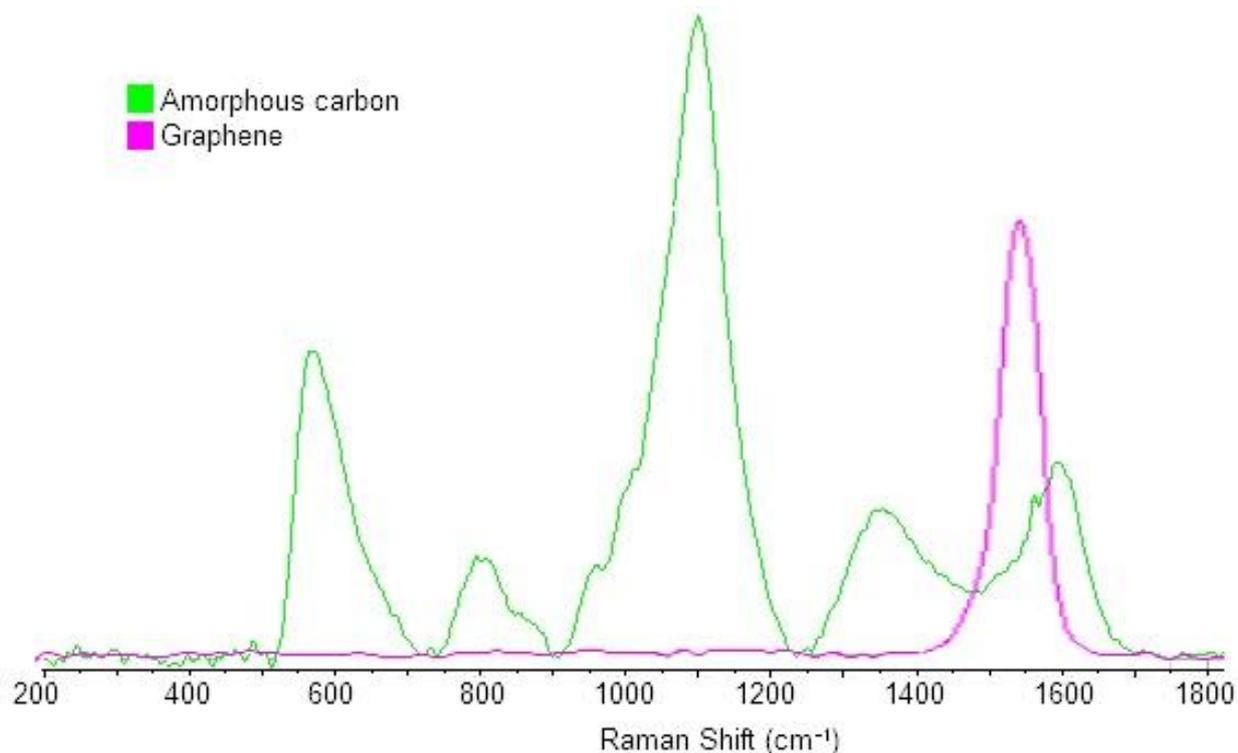
- Heat sinks
- Armour
- Illuminating devices
- Electromagnetic protective skins

Results summary

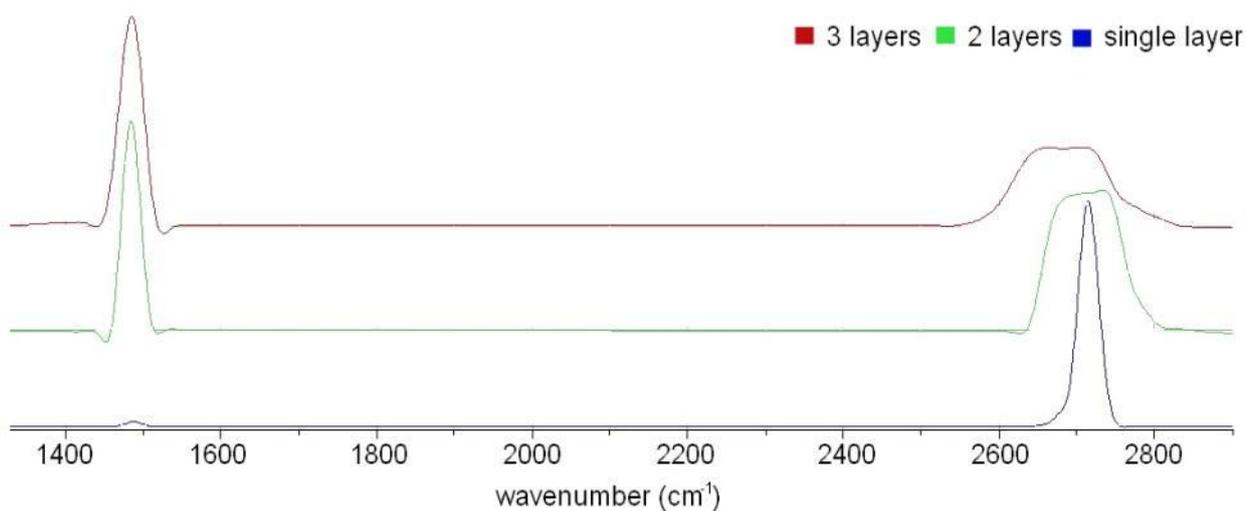
Characteristics of graphene:

- Raman spectroscopy

Graphene can be distinguished from amorphous carbon by its sharp G-band peak which arises from the highly ordered structure. Formation of multiple layers of graphene can be detected by changes in the spectrum, as the peak close to 1500 cm^{-1} grows and the higher peak at 2700 cm^{-1} broadens and decreases.



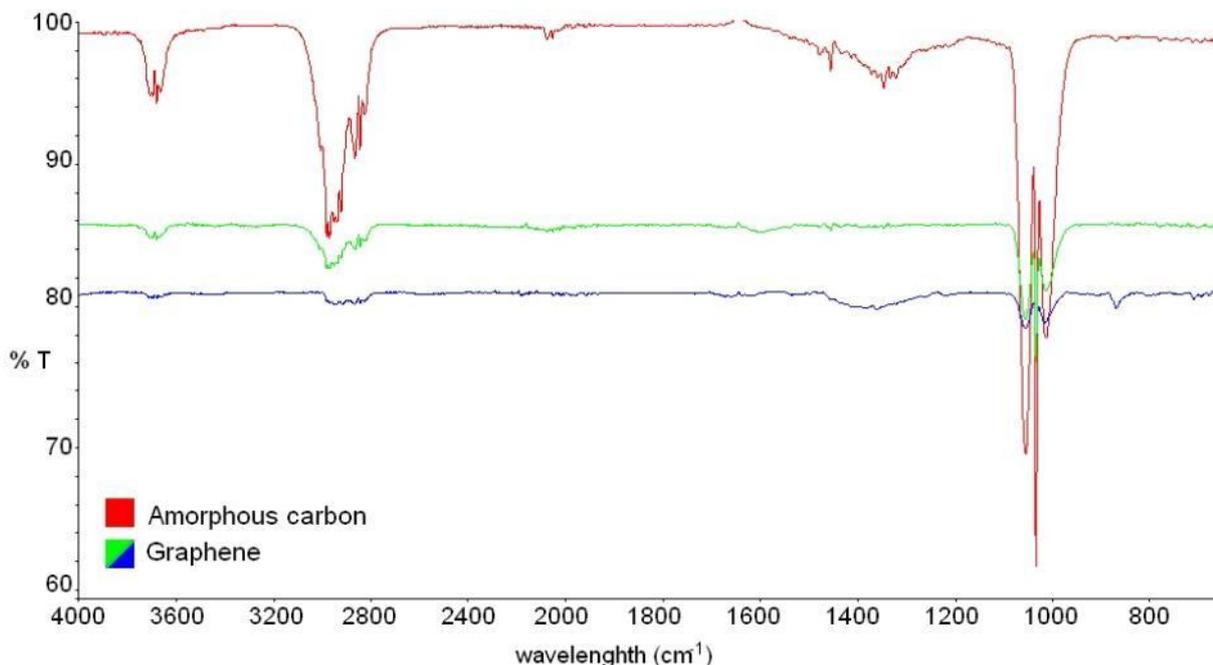
Raman spectrum of amorphous carbon and graphene



Raman spectrum showing layering of graphene

- FTIR spectroscopy

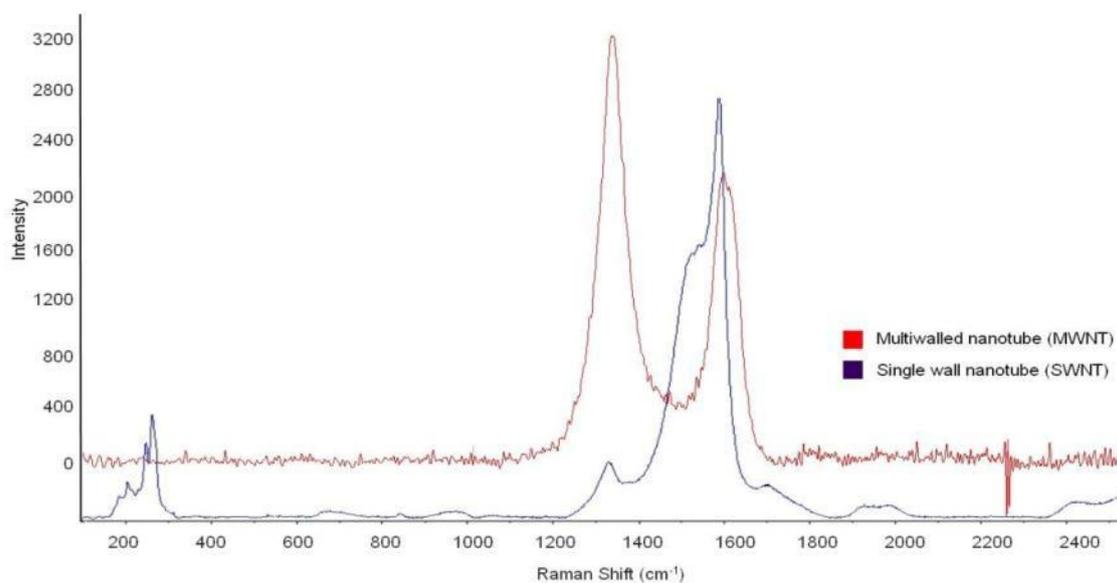
As a pure carbonaceous material, the graphene spectrum does not show the characteristic absorption peaks of impurities in amorphous carbon such as O-H ($\sim 3440\text{ cm}^{-1}$), C-H ($\sim 2900\text{ cm}^{-1}$), and C-O ($\sim 1059\text{ cm}^{-1}$) stretches.



FTIR spectrum of amorphous carbon and graphene

Characteristics of carbon nanotubes (CNTs)

- Raman spectroscopy



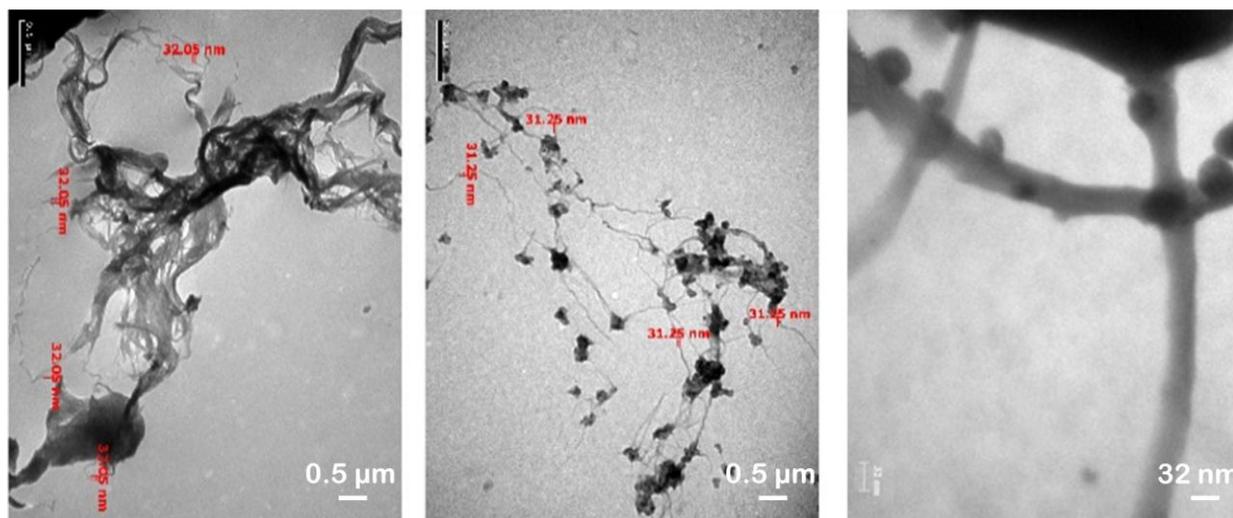
Overlaid Raman spectrum of single and multiwall carbon nanotubes

Single-walled carbon nanotubes (SWNTs) can be prepared by various methods, including pyrolysis of precursor organic molecules, and electrochemical synthesis, while MWNTs are predominantly produced via

arc vaporisation of graphite. As shown above, Raman spectroscopy can be used to distinguish between SWNTs and MWNTs at the carbon defect ($\sim 1300\text{ cm}^{-1}$) and radial breathing mode ($\sim 300\text{ cm}^{-1}$) peaks. The carbon defect peak indicates the presence of amorphous carbon in sample, which will be more abundant in MWNTs due to the increased layers. The radial breathing peak can be correlated with tube diameter. The sharper this peak, the narrower the range of tube diameters present in the sample.

- SEM

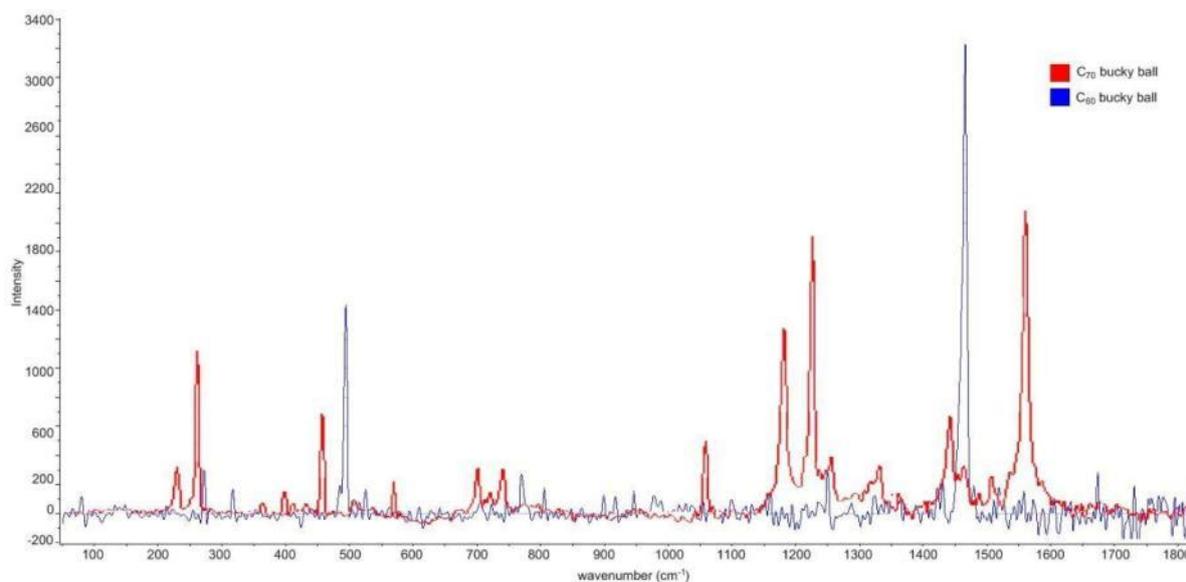
As can be seen by SEM, a variety of different sizes of CNTs may be produced in the same batch. Further treatment is necessary to isolate a narrow range of tube sizes.



Scanning electron microscopy (SEM) image of carbon nanotubes

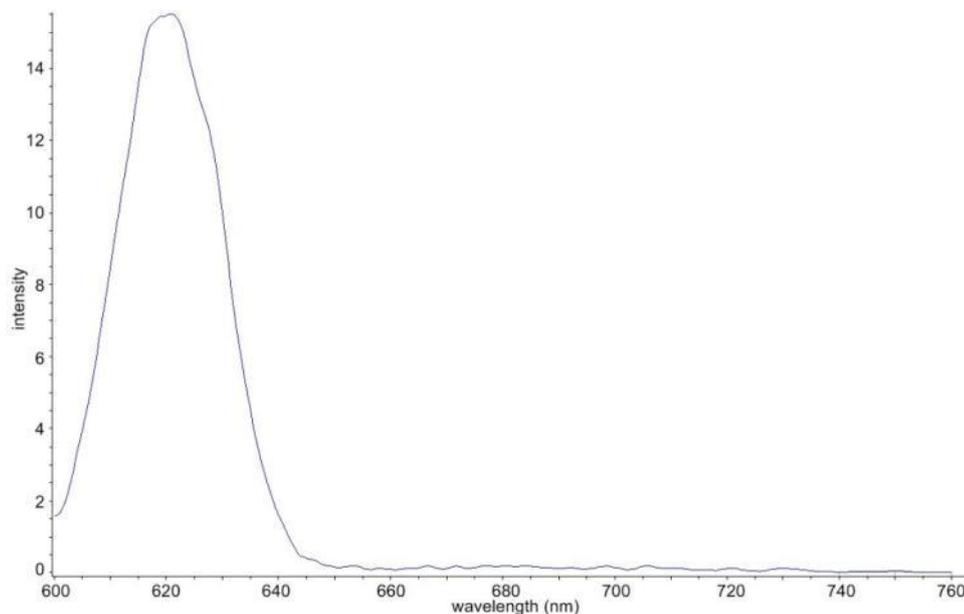
Characteristics of buckminster fullerene balls

- Raman and fluorescence spectroscopy



Raman spectrum of C_{60} and C_{70} molecules

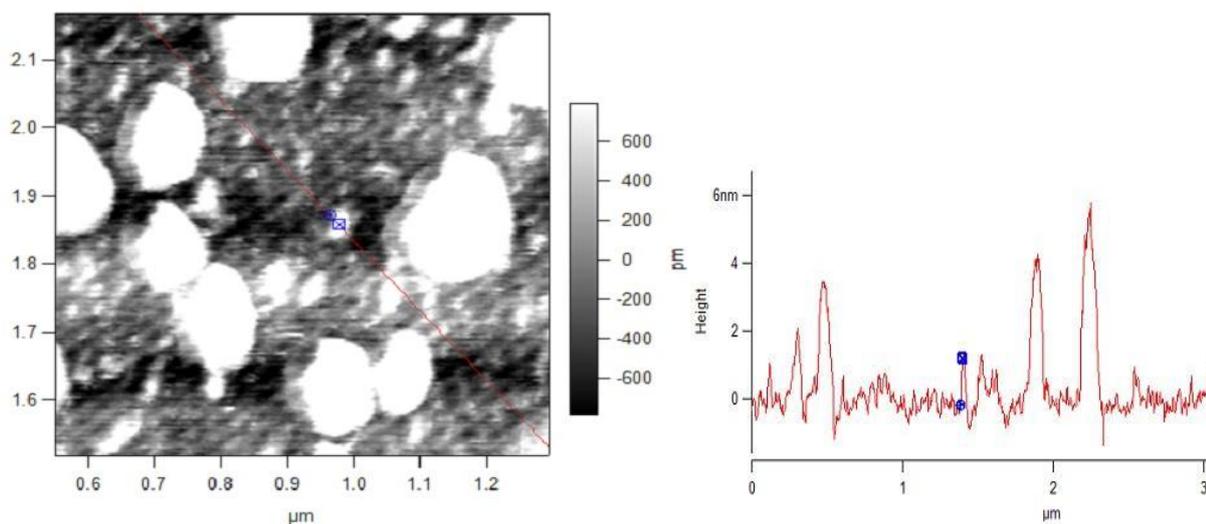
C_{60} buckminster fullerene can be characterised by Raman spectroscopy. The spectrum is much simpler than that of C_{70} due to the symmetry of the molecule and can be used to distinguish it from CNTs or graphene. Bucky balls can also be distinguished from other allotropes as it is the only one which fluoresces.



Fluorescence spectrum of Buckminster fullerene

- Atomic Force Microscope (AFM)

Individual bucky balls have a diameter of 0.7 nm as can be seen by AFM. The figure below shows the measuring of height using AFM. The point highlighted in blue in the image and the corresponding size distribution graph shows a single ball with diameter of 0.7 nm and 1.4 nm as expected. The balls may agglomerate during preparation or storage resulting in formation of larger particles.

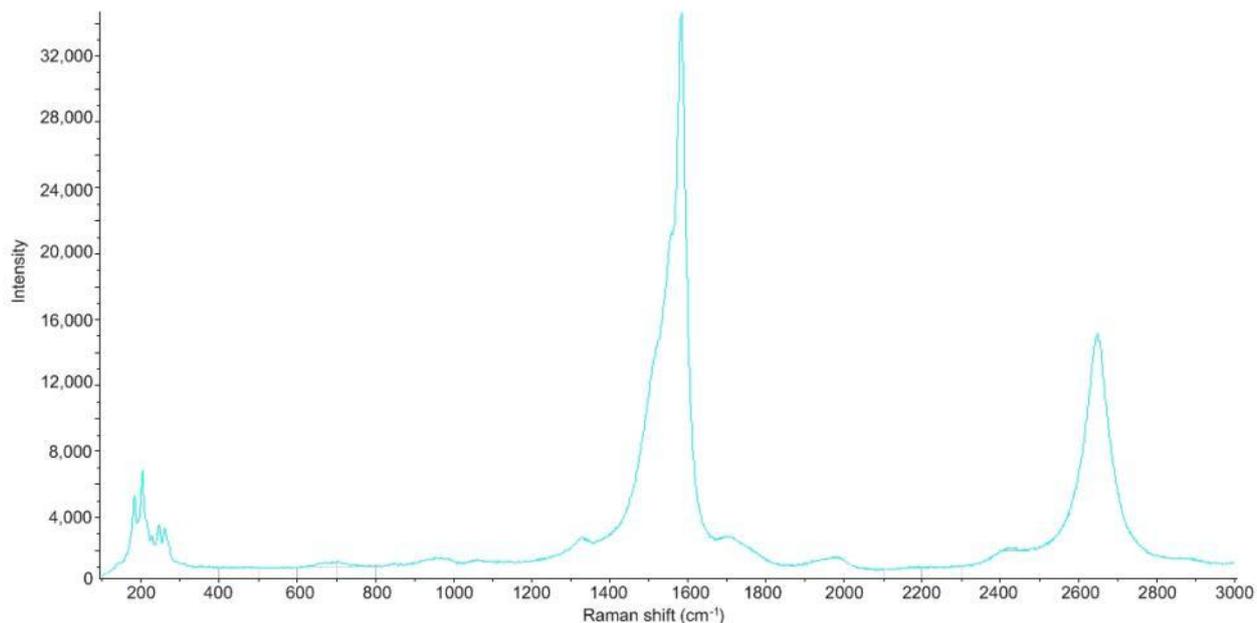


Atomic force microscopy image and associated height measurement for Buckminster fullerene

Characteristics of bucky paper

- Raman spectroscopy

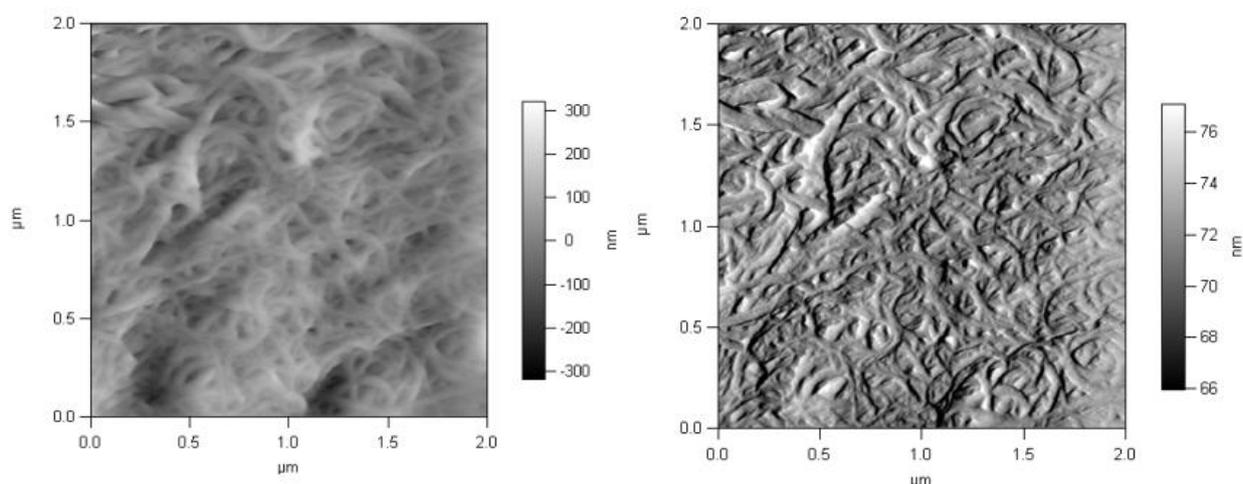
As can be seen in Figure 10, the Raman spectrum of bucky paper is very similar to CNT as it is formed by deposition of tubes on a film. The radial breathing mode ($\sim 300\text{ cm}^{-1}$) may be narrowed as the repeated filtering reduces the range of CNTs deposited. The defect carbon peak ($\sim 1300\text{ cm}^{-1}$) is also reduced by the filtration process as amorphous carbon is removed.



Raman spectrum of bucky paper sample

- Atomic Force Microscopy (AFM)

As the Raman spectrum for bucky balls is so similar to that of CNTs the best method to distinguish from other allotropes is via visual examination (e.g. AFM). The images below show the textured surface of bucky paper which allows clear distinction from CNTs.



Case study 5: Solarsunrayz



Capturing the power of the sun

Solarsunrayz

Solarsunrayz is a research based company that is currently examining the use of Quantum Dots for solar energy capture in solar panels. They wish to explore the types of quantum dots, how they are made and how they may be characterized before use with the relevant costing.

The students are required to prepare a group wiki covering the content of a project description template as shown below.

Project Description Template

Project overview [Abstract/Summary] (max 300 words) Aim of the project. Overview of nanomaterial, including relevant background information.
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Costing How much will the raw materials cost.
Conclusion (max 200 words) Summarise results and recommendations

Quantum dots annotated bibliography

Nanocrystalline Semiconductors: Synthesis, Properties, and Perspectives; Trindade, T., O'Brien, P., Pickett, N.L.; *Chemistry of Materials*, 2001, 13, 3843-3858

Semiconductor nanoparticles promise to play a major role in several new technologies due to their unique chemical and electronic properties. Possible applications include nonlinear optics, luminescence, electronics, catalysis, solar energy conversion, and optoelectronics. The small dimensions of these particles result in different physical properties from those observed in the corresponding "bulk" material, as smaller particle size results in an increase in the ratio of surface atoms to those in the interior, and larger band gap. This allows the opportunity to change the electronic and chemical properties of a material simply by controlling its particle size. This review discusses advances in the synthesis of compound semiconductor nanoparticle materials and their potential uses in areas such as catalysis and electronic device fabrication.

Quantum dot solar concentrators: Electrical conversion efficiencies and comparative concentrating factors of fabricated devices; Gallagher, S.J., Norton, B., Eames, P.C.; *Solar Energy*, 2007, 81, 813-821

This research describes the use of quantum dot technology to produce a fluorescent dye solar concentrator. This class of solar concentrator seeds materials such as plastics and glasses with quantum dots, photovoltaic (PV) cells can then be attached to the edges and converts the direct and diffuse solar energy collected into electricity for use in the building. This article reports production of small scale QDSC devices which were characterised to determine current, voltage and power readings. The electrical conversion efficiencies, fill factors, and comparative concentrating factors are also reported.

Quantum dot containing nanocomposite thin films for photoluminescent solar concentrators; Schuler, A., Python, M., Valle del Olmo, M., de Chambrier, E.; *Solar Energy*, 2007, 81, 1159-1165

CdS quantum dots are incorporated into silicon oxide films deposited on glass substrates in order to grow CdS nanocrystals on the surface. The samples show visible photoluminescence, the colour of which can be tuned by varying the annealing temperature which governs crystal growth and thus the cluster size distribution. The presence of quantum size effects is confirmed in the characteristic features observed in the photoluminescence spectra. The use of these quantum dot containing coatings on glass panes for photoluminescent solar concentrators potentially offers low-cost fabrication on the large scale and the opportunity to be integrated into architectural design.

The use of heat transfer fluids in the synthesis of high-quality CdSe quantum dots, core/shell quantum dots, and quantum rods; Asokan, S., Krueger, K.M., Alkhaldeh, A., Carreon, A.R., Mu, Z., Colvin, V.L., Mantzaris, N.V., Wong, M.S.; *Nanotechnology*, 2005, 16, 2000-2011

Quantum dots have potential uses in a number of optical materials, with advances in chemical synthesis having led to improvements in product control, cost, and safety. Large scale production is limited by the cost of starting materials with, for example, the solvent octadecene accounting for most of the materials cost for a batch of CdSe quantum dots. This paper examines alternative solvents and heat transfer fluids for quantum dot synthesis.

A Safer, Easier, Faster Synthesis for CdSe Quantum Dot Nanocrystals; Boatman, E.M., Lisensky, G.C., Nordell, K.J.; *J. Chem. Ed.*, 2005, 82, 1697-1699

This paper provides a simple synthetic procedure for CdSe quantum dots suitable for application in an undergraduate laboratory.

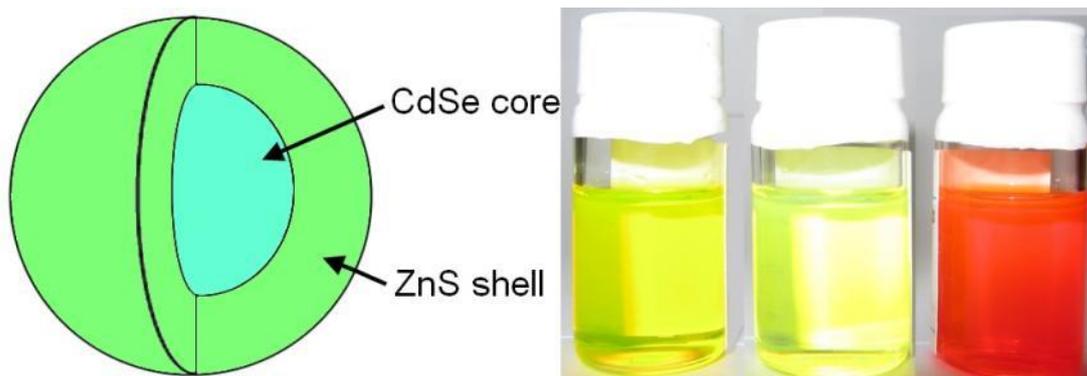
Quantum dots summary report (including preparation and analysis)

Potential applications:

Uses for quantum dots include transistors, solar cells, LEDs, and diode lasers.

Properties:

Three samples of CdSe/ZnS core-shell quantum dots were provided for study. All of the samples, which were produced by Evident Technologies, were suspended in toluene and handled under standard conditions.



Quantum dot structure and samples in toluene (Left to right: Lake Placid Blue, Catskill Green, Fort Orange)

Absorption and emission wavelengths were measured for each sample, followed by emission lifetimes. This information is important when deciding the suitability of the quantum dot for a particular application, e.g. does it absorb visible light? How long does it take to decay and re-emit energy absorbed?

Characterisation of quantum dot samples

Quantum dot	Lake Placid Blue	Catskill Green	Fort Orange
Absorption peak (nm)	461	514	506
Emission peak (nm)	486	538	613
Emission lifetime (ns)	20.21	26.1	19.63
Approximate crystal diameter (nm)	1.9	2.4	4

Note: crystal diameter supplied by manufacturer: Quantum Dot Nanomaterials for Research Specification Sheet, Evident Technologies, Inc., 2006

Appendix 6: Synthetic procedures for preparation of nanomaterials

This section is provided as reference material on the synthesis of the nanomaterials described in this resource. If you wish to incorporate laboratory sessions into the resource, silver, gold and titanium dioxide procedures are suitable for direct application, but the quantum dot synthesis is an advanced practical, suitable only for final year students.

Materials and reagent requirements for laboratory sessions

Table 8 outlines the apparatus needed to complete, each step of the experiment. Standard glassware such as conical flasks, graduated cylinders, and beakers will also be needed at various points throughout the laboratory sessions. These must be very clean before use as residues can cause aggregation of particles,

Table 8: Apparatus requirements

Silver nanoparticle production	Gold nanoparticle production	Titanium dioxide nanoparticle production	CdSe quantum dot production
Clamp and retort stand Well cleaned glassware Burette Stirring plate and magnetic follower Small volume UV-vis cuvettes (plastic can be used)	Clamp and retort stand Stirring hot plate and magnetic follower Small volume UV-Vis cuvettes (plastic can be used)	Clamp and retort stand Burette Stirring plate and magnetic follower High temperature oven in fumehood for calcination	Clamp and retort stand Burette Stirring plate and magnetic follower High temperature oven for calcination

Products are characterised by visible spectroscopy, fluorescence and Raman spectroscopy, XRD, electron microscopy, dynamic light scattering, and luminescent lifetimes.

Table 9 lists the materials and reagents for each synthetic step. Catalogue numbers and prices are given as a guideline only (information valid for Dec 2011).

Table 9: Materials and reagents for laboratory sessions

Chemical	Sigma Aldrich catalogue number	Mass/ volume	Price (excl. VAT)
Silver nanoparticle production			
Silver nitrate (purum p.a., crystallized, ≥99.0%)	85230	25 g	€79.40
Sodium borohydride (powder, ≥98.0%)	452882	25 g	€24.30
Polyvinyl alcohol (PVA) (M _w 89,000-98,000, 99+% hydrolyze)	341584	25 g	€34.40
Tri sodium citrate (TSC) (≥98% purum p.a., ≥ 99.0%)	C7254	1 kg	€41.50
Hydrazine, (reagent grade, 50-60 %)	225819	100 mL	€29.30
Gold nanoparticle production			
Hydrogen tetrachloroaurate trihydrate (ACS Reagent, > 49%)	G4022	1 g	€102
Sodium citrate dehydrate (ACS Reagent, >99%)	S4641	25 g	€27
Titanium dioxide nanoparticle production			
Titanium(IV) isopropoxide(97%,	205273	100 mL	€33.81
Urea (ACS Reagent, 99 %,	U5128	100 g	€19.40
glacial acetic acid (ACS Reagent, >99.7%)	695092	100 mL	€12
Isopropanol (ACS Reagent, 99 %,	673773	1 L	€65.50
CdSe quantum dot production			
Selenium (powder, 100 mesh, 99.5+%,	209651	50 g	€32.30
1-Octadecene (tech. 90%,	O806	25 mL	€17.20
Trioctylphosphine (tech. 90%,	117854	25 mL	€46
Cadmium oxide (~1 micron, 99.5%,	244783	100 g	€40
Oleic acid (tech. 90%,	364525	1 L	€38.40

Preparation of Silver Nanoparticles

Safety Information

Substance name	CAS no	Hazard Classification	Hazard statement/Risk phrase	Route of exposure	OELV ¹
Silver nitrate 	7761-88-8	Causes burns. Very toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment. Contact with combustible material may cause fire. DANGER	H272 May intensify fire; oxidiser. H314 Causes severe skin burns and eye damage. H410 Very toxic to aquatic life with long lasting effects.	Skin contact, Inhalation	8 hr: 0.01 mg/m ³
Sodium Borohydride 	16940-66-2	Contact with water liberates extremely flammable gases. Toxic in contact with skin and if swallowed. Causes burns. DANGER	H260 In contact with water releases flammable gases which may ignite spontaneously. H301 Toxic if swallowed. H311 Toxic in contact with skin. H314 Causes severe skin burns and eye damage.	Skin contact, Inhalation	
Poly Vinyl Alcohol	9002-89-5	Not a dangerous substance according to GHS.	n/a	Skin contact, Inhalation	
tri Sodium Citrate	6132-04-3	Not a dangerous substance according to GHS.	n/a	Skin contact, Inhalation	
Hydrazine Hydrate (N ₂ H ₄) 	10217-52-4	Toxic by inhalation, in contact with skin and if swallowed. May cause cancer. Causes burns. May cause sensitization by skin contact. Very toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment. DANGER	H301 Toxic if swallowed. H312 Harmful in contact with skin. H314 Causes severe skin burns and eye damage. H317 May cause an allergic skin reaction. H331 Toxic if inhaled. H350 May cause cancer. H410 Very toxic to aquatic life with long lasting effects.	Skin contact, Inhalation	8 hr: 0.01 mg/m ³

OELV = occupational exposure limit as set down in the most up to date Code of Practice for the Chemical Agents Regulations.

Technical notes:

- Check that all glassware is very clean; impurities on the glassware can affect results.
- Use distilled deionised water to make up all solutions.
- Most of the stock solutions prepared last several months once stored in a cool dark place.
- All of the stock solutions are clear and colourless.
- Sodium borohydride decomposes in water; therefore it should be prepared fresh before use, keeping the solution in ice water will reduce the rate of decomposition and prolong its shelf life. Do not stopper this solution tightly as pressure is likely to build up in the vessel.
- While polyvinyl alcohol (PVA is water soluble it takes several hours to completely dissolve. Best results were observed by stirring the PVA in distilled water at 80 °C for 2-3 hours to ensure full dissolution. It is important to use the correct grade of PVA (M_w 89,000-98,000) as others may not dissolve fully.
- Hydrazine can be found readily only as a solution; in this case 50 - 60%, this should be taken into account when preparing solutions.
- Silver nitrate solution should be stored in a dark glass container to prevent degradation. Gloves should be worn when handling silver nitrate solution to prevent staining of skin.
- For best results the addition of the 0.001 M silver nitrate should be drop wise with constant stirring.
- It may be helpful for students to be shown the synthesis portion of the silver nanoparticle electronic resource before the laboratory session. It is useful for them to see what colour changes to expect so they know if their experiment is going right.

Procedure:

Seed production:

1. Add 2 cm³ 0.001 M AgNO₃ to 2 cm³ of PVA (1% w/v) and mix well.
2. While stirring constantly, add 2 cm³ dropwise of 0.001 M NaBH₄ to this solution until the solution goes golden yellow.
3. The resulting solution should be a golden yellow, if the solution goes black too much borohydride has been added.

Fabrication of coloured colloids:

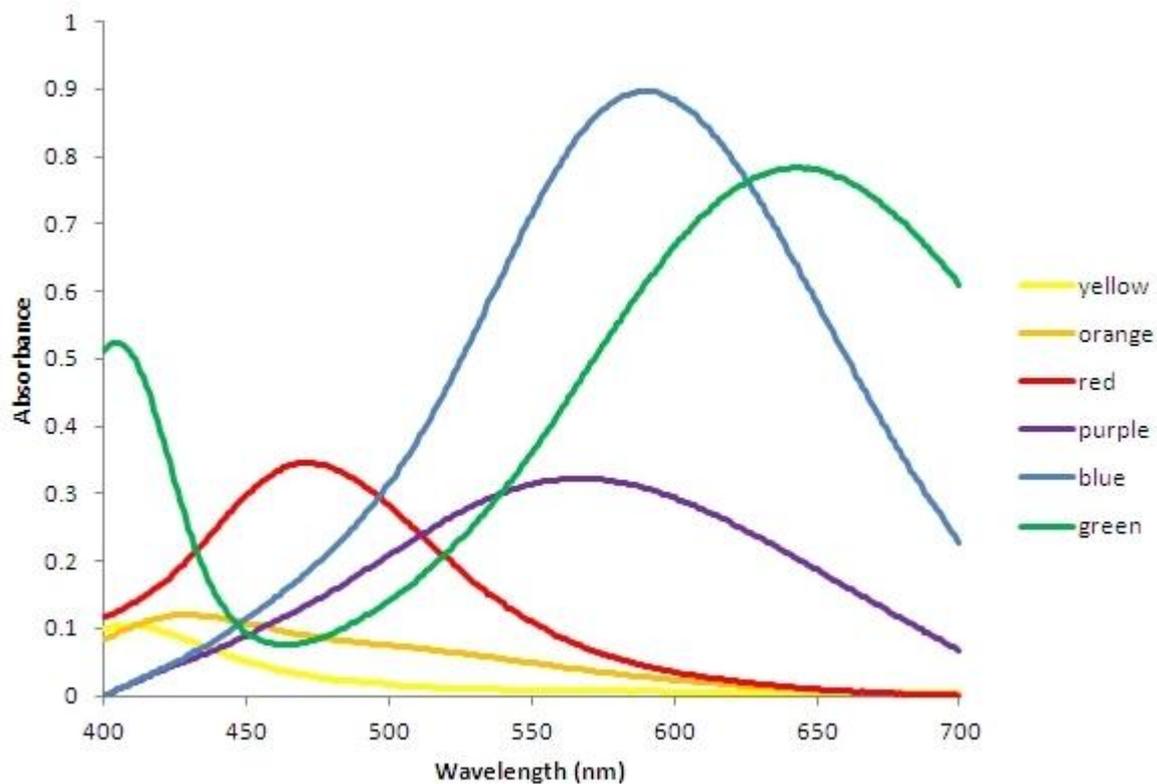
4. Add 1 cm³ of the seed solution, 3 cm³ 0.1 M tri sodium citrate (TSC), and 5 cm³ 0.1 M hydrazine (H₄N₂) to 1 cm³ PVA (1% w/v) making sure to stir well.
5. Produce coloured colloids by addition of 0.001 M AgNO₃ dropwise, while continuing stirring of this 'growth mixture'.
6. A first titration style run should be done to determine volumes of AgNO₃ required to prepare specific coloured colloid solutions. See table one for sample results.
7. With the information collected in step 3, colloid solutions should be prepared by addition of known volumes of AgNO₃ to portions of the growth mixture.

Visible spectroscopy analysis:

Samples should be prepared for analysis by diluting in distilled water in the ratio of 1:3. Spectra are measured between 400 and 700 nm, using water as a reference. Overlay the spectra for best presentation of results.

Results:

Colloid colour	Volume AgNO ₃ added (cm ³)	Vis λ max (nm)
Yellow	0.2	411
Orange	0.4	428
Red	1.2	471
Purple	2.8	568
Blue	8	592
Green	20	404, 644



Preparation of Gold Nanoparticles²

Safety Information

Substance name	CAS no	Hazard Classification	Hazard statement/Risk phrase	Route of exposure	OELV
Gold(III) chloride trihydrate 	16961-25-4	Causes burns. May cause sensitization by skin contact. DANGER	H314 Causes severe skin burns and eye damage. H317 May cause an allergic skin reaction.	Skin contact, Inhalation	
tri Sodium Citrate	6132-04-3	Not a dangerous substance according to GHS.	n/a	Skin contact, Inhalation	

Technical notes:

- Unused Au nanoparticle solution made can be stored for several years in a brown glass bottle, while 1.0 mM HAuCl₄ stock solution is unstable and will last only a few days.
- Electrostatic repulsion effects within the colloid solution can be studied by adding a few drops of 1M NaCl solution. This will cause the particles to aggregate and fall out of solution.

Procedure

1. Prepare stock solutions of 1.0 mM HAuCl₄ and 38.8 mM Na₃C₆H₅O₇ (sodium citrate) in distilled water.
2. Heat 20 cm³ of 1.0 mM HAuCl₄ to boiling while stirring with the magnetic stir bar.
3. As the solution begins to boil, add 2 cm³ of the 38.8 mM sodium citrate solution, continuing to boil and stir the solution until it is a deep red color (about 10 min). Add distilled water as needed to keep the total solution volume constant at 22 cm³.
4. When the solution is a deep red colour, turn off the hot plate and stirrer. Cool the solution to room temperature.

Preparation of Titanium Dioxide Nanoparticles³

Safety Information

Substance name	CAS no	Hazard Classification	Hazard statement/Risk phrase	Route of exposure	OELV
Titanium(IV) isopropoxide 	546-68-9	Flammable. Harmful by inhalation. Irritating to eyes. DANGER	H226 Flammable liquid and vapour. H319 Causes serious eye irritation. H331 Toxic if inhaled.	Skin contact, Inhalation	
Acetic acid 	64-19-7	Flammable. Causes severe burns. DANGER	H314 Causes severe skin burns and eye damage. H226 Flammable liquid and vapour.	Skin contact, Inhalation	8 hr: 25 mg/m ³

Technical notes:

- Ensure to follow order given in procedure. If titanium isopropoxide is added directly to water, the solution will solidify and become unworkable.
- Set solution stirring speed to the maximum practicable speed as even in the presence of acetic acid the solution may solidify.
- If the solution does solidify, while following the procedure correctly, it should be possible to get it to dissolve again by stirring with a spatula.

Procedure

1. Add 25 cm³ of 90 mM titanium isopropoxide to 48 cm³ of 0.8 M glacial acetic acid stirring on a magnetic stirrer plate.
2. Add 150 cm³ of deionised water drop-wise to the partially hydrolysed titanium isopropoxide-acetic acid mixture. The solution will turn white. Ensure to maintain vigorous stirring.
3. Stir the solution until a clear transparent solution or sol is formed; this may take up to 8 hours.
4. Dry overnight at 100 °C in an oven vented into a fumehood.
5. Calcine the final product in air for 2 hours at 600 °C (anatase phase) and 700 °C (rutile phase) (ramp rate 5 °C min⁻¹).

Preparation of N-doped Titanium Dioxide Nanoparticles⁴

Safety Information

Substance name	CAS no	Hazard Classification	Hazard statement/Risk phrase	Route of exposure	OELV
Titanium(IV) isopropoxide 	546-68-9	Flammable. Harmful by inhalation. Irritating to eyes. DANGER	H226 Flammable liquid and vapour. H319 Causes serious eye irritation. H331 Toxic if inhaled.	Skin contact, Inhalation	
Urea	57-13-6	This substance is not classified as dangerous.		Skin contact, Inhalation	
Isopropanol 	67-63-0	Highly flammable. Irritating to eyes. Vapours may cause drowsiness and dizziness. DANGER	H225 Highly flammable liquid and vapour. H319 Causes serious eye irritation. H336 May cause drowsiness or dizziness.	Skin contact, Inhalation	8 hr: 200 ppm

Technical notes:

- Ensure to follow order given in procedure. If titanium isopropoxide is added directly to water, the solution will solidify and become unworkable.
- Set solution stirring speed to the maximum practicable speed as even in the presence of acetic acid the solution may solidify.
- If the solution does solidify, while following the procedure correctly, it should be possible to get it to dissolve again by stirring with a spatula.

Procedure:

1. Add 23.4 cm³ of 90 mM titanium isopropoxide to 206 cm³ of isopropanol stirring on a magnetic stirrer plate.
2. Add 5 g of urea dissolved in 35 cm³ of distilled water. Ensure to maintain vigorous stirring.
3. Stir the solution for 5 minutes.
4. Dry overnight at 80 °C.
5. Calcine the final product in air for 2 hours at 400 °C (ramp rate 5 °C min⁻¹).

Preparation of CdSe Quantum Dot Nanocrystals⁵

Safety Information

Substance name	CAS no	Hazard Classification	Hazard statement/Risk phrase	Route of exposure	OELV
Selenium 	7782-49-2	Toxic by inhalation and if swallowed. Danger of cumulative effects. May cause long-term adverse effects in the aquatic environment. DANGER	H373 May cause damage to organs through prolonged or repeated exposure. H301 Toxic if swallowed. H331 Toxic if inhaled. H413 May cause long lasting harmful effects to aquatic life.	Skin contact, Inhalation	8 hr: 0.1 mg/m ³
1-octadecene 	112-88-9	Harmful: may cause lung damage if swallowed. May cause long-term adverse effects in the aquatic environment. DANGER	H304 May be fatal if swallowed and enters airways.	Skin contact, Inhalation	
Trioctylphosphine 	4731-53-7	Causes burns. DANGER	H314 Causes severe skin burns and eye damage.	Skin contact, Inhalation	
Cadmium oxide 	1306-19-0	May cause cancer. Possible risk of irreversible effects. Very toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment. Possible risk of impaired fertility. Possible risk of harm to the unborn child. Very toxic by inhalation. Toxic: danger of serious damage to health by prolonged exposure through inhalation and if swallowed. DANGER	H330 Fatal if inhaled. H341 Suspected of causing genetic defects. H350 May cause cancer. H361 Suspected of damaging fertility or the unborn child. H372 Causes damage to organs through prolonged or repeated exposure. H410 Very toxic to aquatic life with long lasting effects.	Skin contact, Inhalation	Fume: 8 hr: 0.025 mg/m ³
Oleic acid 	112-80-1	Irritating to skin. WARNING	H315 Causes skin irritation.		

Procedure

1. Prepare a 0.038 M stock solution of Se precursor by adding 30 mg Se and 5 cm³ 1-octadecene (tech., 90%) in a 10 cm³ round bottom flask clamped over a stirrer hot plate.
2. Using a syringe add 0.4 cm³ trioctylphosphine from its Sure-Seal container. Stir the solution on a stirrer plate. Warm the solution as necessary to speed dissolution of the Se. This stock solution can be stored at room temperature in a sealed container and has enough Se precursor for five preparations. The system can be scaled up as required.
3. Using a balance in an extractor fumehood weigh out 13 mg CdO.
4. Add the CdO to a 25 cm³ round bottom flask with 0.6 cm³ oleic acid and 10 cm³ octadecene.
5. Clamp in a heating mantle and insert a thermometer capable of measuring to 225 °C.
6. Prepare 10 test tubes in a rack.
7. When the temperature reaches 225 °C, add 1 cm³ of the room temperature selenium stock solution to the cadmium solution. Start timing when the selenium solution is added as the size of the nanoparticles depends on reaction time.
8. Rapidly remove approximately 1 cm³ samples at frequent intervals into separate test tubes. Nine or ten samples should be collected within two to three minutes.
9. Record the UV-Vis absorption and emission spectra of individual samples to find maximum wavelength peaks. An excitation wavelength of 400 nm can be used for all emission samples

For (CdSe)ZnS core shell preparation see Dabbousi et al J.Phys Chem 1997⁶ although this is quite a complex procedure, which may be difficult to repeat in an undergraduate lab.

Appendix 7: Full length urls

The urls used in this guide have been shortened for ease of use. In the case where the shortened urls no longer work the list below provides the original.

<http://tinyurl.com/nanochem1> <http://royalsociety.org/Event.aspx?id=8417&gclid=CPCg9-bQ5aoCFQkf4QodIFbU6A>
2 <http://www.rsc.org/chemistryworld/Issues/2009/January/FeynmansFancy.asp>
3 <http://www.evidenttech.com/technology/basic>
4 <http://www.trynano.org>
5 <http://www.rsc.org/chemistryworld/Issues/2005/December/nano.asp>
6 <http://www.rsc.org/chemistryworld/News/2009/February/25020901.asp>
7 http://www.rsc.org/images/Nanomaterials_tcm18-187860.pdf
8 http://www.nanowerk.com/nanotechnology/ten_things_you_should_know_about_nanotechnology.php
9 <http://nanosense.org>
10 <http://mrsec.wisc.edu/Edetc/nanolab/>
11 <http://www.nanotechproject.org/topics/nano101/>
12 <http://community.acs.org/nanotation/>
13 <http://research.uiowa.edu/nniui/links.html>
14 <http://youtu.be/e80bflcoNUA>
15 <http://education.techyou.edu.au/field>
16 <http://youtu.be/XurpzMGRRnU>
17 <http://youtu.be/1GFst2IQBEM>
21 www.edrawsoft.com/freemind.php
22 www.visual-mind.com/download.php
24 <http://iwawaterwiki.org/xwiki/bin/view/Articles/tonni6>
25 <http://future.wikia.com/wiki/Nanotechnology>
26 <http://nanowiki.info/>
29 <http://usermanual.pbworks.com/w/page/11632091/Glossary>
30 <https://plans.pbworks.com/academic>
31 <http://usermanual.pbworks.com/w/page/11632089/Home>
32 <http://www.youtube.com/watch?v=gRj5ABJ-IPY&feature=related>
33 http://online.cofa.unsw.edu.au/sites/default/files/episode-pdf/CS_Wikis_LTTO.pdf
34 <http://www.youtube.com/watch?v=SZ5OV14v4xU>
35 <https://blog.itu.dk/MVOL-F2010/files/2010/02/corporate-wiki-users-results-of-a-survey.pdf>
36 <http://cagreenchem.wikidot.com/start>
37 <http://cagreenchem.wikidot.com/welcome>
38 <http://eponline.com/articles/2009/02/09/calif-launches-wiki-to-develop-green-chemistry-regulations.aspx>
39 http://www.ganfyd.org/index.php?title=Main_Page
40 http://www.rsc.org/images/Guidelines_tcm18-186308.pdf
41 <http://en.wikipedia.org/wiki/Nanochemistry>
42 <http://en.wikipedia.org/wiki/Nanoparticles>
43 <http://www.nanopaprika.eu/profiles/blogs/research-in-nanotechnology-to>
44 <http://health.howstuffworks.com/medicine/modern-technology/gold-nanotech.htm>
45 <http://science.howstuffworks.com/nanotechnology5.htm>
46 <http://www.dailymail.co.uk/sciencetech/article-1218300/Nano-magnets-seek-destroy-cancer-cells-developed-British-experts.html>

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