Learn Chemistry

Pollutant Detection and Remediation

Tutor Guide

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Table of Contents

This Tutor Guide accompanies the resource "*Pollutant Detection and Remediation*", a context and problem-based learning resource developed for the Royal Society of Chemistry in 2012 as part of the HE-STEM programme. It is intended to accompany a physical chemistry/materials chemistry curriculum at an intermediate level.

Introduction to Context and Problem Based Learning

The resource differs from a traditional laboratory course in several ways. Firstly, it is a **context-based case study**, so that students can relate the experiments being conducted to a real-world application. The context is the development of remediation strategies for a polluted water pond at a fish farm in the fictional county of Fercullen. It is based on real media reports of the use of malachite green as a fungicide in aquaculture, which despite being illegal because of toxicity, is still used because of low cost and effectiveness. Students act as scientific consultants working for Whitewater Environmental Services, who are requested to validate a method for quantifying the amount of malachite green in a sample, develop methods for removal of malachite green from water in an environmentally sustainable manner, and use their experimental evidence to recommend a remediation strategy to the Council.

The second difference between this resource and a traditional laboratory approach is the use of a **problem-based approach**. This means that the laboratory sessions do not provide students with direct instructions requiring them to develop their own experimental protocols. Guidance on how to do this is provided and the sessions are arranged so that the experimental protocols become progressively more complex, allowing students to develop this skill over the course of completing the resource. Literature references (compiled list in Appendix) are provided to help students source relevant information to develop experimental protocols and outline protocols are provided in this guide. Requiring students to develop protocols means that more laboratory time is needed to allow students to pilot experimental techniques. This is built into the schedule of implementation. While this approach takes more time to complete than traditional laboratory activities, the intention is that as well as becoming proficient in the relevant laboratory techniques, students gain more experience in information retrieval and application, laboratory planning, and trouble-shooting than they might with a traditional laboratory teaching method.

Thirdly, the resource exposes the student to meaningful **group work**, both within their assigned group and as a class. Students work in small teams with guidance provided on how to complete each session's activities, before, during, and after the weekly contact session. In addition, several of the laboratory sessions offer the opportunity to share results across the class, so that students can formally compare and contrast their experimental data to others and draw conclusions from this. This approach aims to provide a greater sense of motivation in that the students are completing activities to contribute to the greater body of knowledge required to complete the project, rather than just "get the right answer", which is a common student conception of traditional laboratory classes.

Finally, the resource uses an online document building environment called a **wiki** to collaboratively build a report over the course of the case study. This allows all students to contribute to the final report, and allows the tutor monitor progress and contributions of both groups and individuals as the resource is being developed. Extensive guidelines for wikis are provided in the Appendix to this *Tutor Guide*. A brief synopsis follows.

A word about wikis

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 Lever Arch file 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 this draft work and supporting information is submitted as an appendix to the final report.

Resource at a Glance

The resource is a laboratory-based case study involving five core laboratory sessions, and ancillary preparatory and feedback sessions. Opportunities to extend the resource if more time is available or to curtail it to a smaller number of sessions are highlighted in the *Navigating the Case study* section below. In completing the resource, students will:

- Use UV/visible spectroscopy to quantify the amount of malachite green present.
- Study the effect of physical adsorption as a remediation strategy.
- Study the effect of photocatalysis as a remediation strategy.

Navigating the Resource

This case study resource consists of 8 three hour sessions designed to fit into a laboratory schedule at an intermediate level chemistry degree. As such it represents approximately 2.5 European Credit Transfer System (ECTS) or 5 UK credits of work or 1.5 US credits (based on 24 contact hours, 26 self study). The resource includes an induction session, five laboratory sessions, a feedback session and a presentation session. Information on planning and running each of these sessions is provided in this *Tutor Guide*, with parallel information for students provided in the *Student Guide*. Each session outlines what to do before, during and, for the student, after each session. For the tutor, a schedule of what feedback/prompts/actions may be required is shown in Table 2, aligned with the requirements of the students.

While the resource has been designed as an entire unit, it may be desirable to just use one or a few components, or to extend the implementation of the resource beyond the scope described here. Some brief comments on each session follow so that tutors may decide whether there is scope to limit/extend the resource to suit academic requirements. Of course in limiting the implementation, some of the learning outcomes may not be achieved.

Session 1: This is an induction session in a classroom setting. The purpose of the session is to introduce students to the resource and begin the experimental planning with the support of the tutor (in future sessions this planning will be done in the students' own time).

Session 2: This session is a laboratory session where the students have to implement their first experimental protocol. The subject of the laboratory work (Beer-Lambert Law) is purposefully easy, as feedback has demonstrated that there is an initial steep learning curve for students given freedom to implement their own laboratory procedures (i.e. without a 'recipe' provided). Tutors whose students are at a more advance stage or with experience in less expository-based practical classes may wish to incorporate some information here on water sampling to link in with the context, or provide students with an unknown sample to test the calibration data.

Sessions 3 and 4: These sessions, both laboratory sessions, are about designing and implementing an adsorption study for the model pollutant onto surfaces. In the first session, students become familiar with the Langmuir analysis through a laboratory using filter paper. Guidance notes from the literature are provided. This is followed in the second session with a similar protocol for another adsorbent – titanium dioxide is the example illustrated here. It may be that students already have experience in a surface-chemistry laboratory of this nature, and may not need two sessions. Those wishing to extend the resource are pointed to a review of low-cost adsorbents for the purpose described in this case study, which would allow for substantial data gathering to be implemented by several students. These sessions are the most challenging in terms of data analysis.

Sessions 5 and 6: These sessions, both laboratory sessions, are about designing and implementing a photocatalysis study for the degradation of the model pollutant. Photocatalysis is not a common laboratory in physical chemistry curricula, but is one of a growing number of advanced oxidation processes (AOPs) being considered for environmental remediation. Again, those wishing to limit the number of sessions could provide more guidance or limit the number of parameters to be investigated, so as to reduce it to one session. Those looking to extend it are provided with several parameters for investigation. Advance planning is required here in terms of sourcing suitable photocatalytic materials.

Session 7: This is a feedback clinic, which aims to give the students some interim feedback on their final report. If time is limited, this could be completed electronically via the virtual learning environment.

Session 8: This is a presentation session where students present their work. Again, if time is limited, the assessment could be modified and the presentation omitted.

Assessment

The resource contains three main elements: planning laboratory experiments, conducting experiments and analysis and reporting of data obtained. Therefore, assessment focuses on:

1. The planning of the group project: most conveniently monitored using a wiki.
2. The conducting of experiments: Student laboratory books that should record experiments conducted.
3. The reporting: analysis and recommendations arising from the data obtained.

More details on assessment are provided at appropriate points in this guide and the assessment components and a guideline weighting are provided in Table 1 (also used in the Student Guide (Table 1) and introductory presentation).

Table 1: Assessment components

Activity		% mark allocation (guideline)
Participation in laboratory and workshop sessions and contribution to group wiki *You may choose a component of this mark (e.g 5%) to be peer assessment by other group members (frequency and quality of contributions, both online and face-to-face)*	Individual	15
Laboratory notebook	Individual	25
Reflective piece	Individual	10
Final wiki report (Criteria - content, accuracy, structure, clarity, references to the literature)	Group	30
Presentation (assessment by tutor, feedback from tutor and peers, peer assessment is optional)	Group	20

Implementation: Class organisation

The resource is designed so that students work in small groups (typically three students) to complete the brief provided. The assessment components (Table 1) require that each student submits a laboratory book as well as a short reflective summary for individual assessment. The assessed group work covers the presentation and report and the group wiki, which provides a record of how the group collaborated and their rate of progress. The report can be generated directly from the wiki either by converting wiki pages to PDF files or by printing the pages (depending on the software used). Advice on monitoring and correcting wikis is provided in this guide, but, in short, the tutor should log into each group's wiki approximately once per week if possible (assuming 3 hours contact per week), and provide brief feedback on progress reported. Feedback is also provided on any components of the group report that are submitted in a given session (e.g. experimental graphs from data), and the draft group reports are reviewed before Workshop 7. The remaining correction time is spent on the presentation and the completed reports. In this manner, students receive feedback at various stages throughout the process. A suggested schedule of work to be submitted by students and the suggested feedback provided is shown in Table 2, but it will be at a tutor's discretion to decide the extent of the feedback that it is reasonable for them to provide. The assignment of tasks as individual or group activities can also be adjusted as the tutor feels is appropriate. The work is designed so that students are preparing elements of their final report as they go along. The tutor workload aims to be similar to that required in running a traditional laboratory programme (e.g. correction of laboratory reports).

Table 2: Work to be completed after each session & associated suggested feedback

(Suggested comments for different levels of feedback in the wiki are provided in the appendix)

Session	Work Required	Suggested feedback
Week 1 Date:	**Group** • Experimental procedure for Session 2 (Beer-Lambert Law) with chemical safety assessment. • Summary of group meeting posted on group wiki. **Individual** • Contribute some information on background to Session 2 to *Project Introduction* section of wiki in preparation for final presentation. • Maintain independent laboratory book and group wiki.	Any required changes. Acknowledgement. Brief comment to group on wiki. Brief comment on wiki. Check to see everyone has logged in and contributed. Sign and date laboratory book at next session.
Week 2 Date:	**Group** • Summary of data obtained from Session 2 with associated graphs. • Compilation and sharing of group results from Session 2 experiments with other groups. • Experimental procedure for Session 3 (Adsorption Experiments I) with chemical safety assessment. • Summary of group meeting posted on group wiki. **Individual** • Contribute some information on background to Session 3 to *Project Introduction* section of wiki in preparation for final presentation • Maintain independent laboratory book and group wiki.	Brief comment on wiki. Acknowledgement Any required changes. Acknowledgement. Brief comment to group on wiki. Brief comment on wiki, sign and date laboratory book at next session.
Week 3 Date:	**Group** • Summary of data obtained from Session 3 with associated graphs. • Experimental procedure for Session 4 (Adsorption Experiments II) with chemical safety assessment. • Complete background to adsorption experiments to *Project Introduction* section of wiki in preparation for final presentation. • Summary of group meeting posted on group wiki. **Individual** • Maintain independent laboratory book and group wiki.	Brief comment on wiki. Any required changes. Acknowledgement. Brief comment on wiki. Brief comment on wiki, sign and date laboratory book at next session.
Week 4 Date:	**Group** • Summary of data obtained from Session 4 with associated graphs. • Compilation and sharing of data on group results for adsorption experiments with other groups. • Experimental procedure for Session 5 (Photocatalysis I) with chemical safety assessment. • Summary of group meeting posted on group wiki.	Brief comment on wiki. Acknowledgement. Any required changes. Acknowledgement.

	Individual • Contribute some information on background to Session 5 to *Project Introduction* section of wiki in preparation for final presentation • Maintain independent laboratory book and group wiki.	Brief comment to group on wiki. Brief comment on wiki, sign and date laboratory book at next session.
Week 5 Date:	**Group** • Summary of data obtained from Session 5 with associated graphs. • Experimental procedure for Session 6 (Photocatalysis II) with chemical safety assessment. • Complete background to photocatalysis experiments to *Project Introduction* section of wiki in preparation for final presentation. • Summary of group meeting posted on group wiki. **Individual** • Maintain independent laboratory book and group wiki.	Brief comment on wiki. Any required changes. Acknowledgement. Brief comment on wiki Brief comment on wiki, sign and date laboratory book at next session.
Week 6 Date:	**Group** • Compilation and sharing of data on group results for photocatalysis experiments with other groups. • Analysis of results for both remediation strategies across various parameters studied by all groups. • Summary of group meeting posted on group wiki. • Work in progress draft report ("interim report") posted on group wiki. **Individual** • Maintain independent laboratory book and group wiki.	Acknowledgement. Acknowledgement. Acknowledgement. Review before next session and prepare a short summary of the aspects dealt with well and those that need more work. Check to see if anyone has not been contributing at all. Brief comment on wiki.
Week 7 Date:	**Group** • Incorporation of feedback from clinic workshop into the group's wiki report. • Discussion of the data obtained from group and class experiments for each method (adsorption and photocatalysis). • Practise group presentation to ensure it is coherent, structured, accurate and meets the time requirements (the wiki itself can be used as a visual aid or, alternatively, you may prefer that PowerPoint slides be prepared). • Summary of group meeting posted on group wiki. • Maintain group wiki.	Any required changes. Provide oral and brief written feedback after presentation at next session. Acknowledgement. Brief comment on wiki.
Week 8 Date:	**Group** • Incorporation of feedback from presentation into the group's wiki report. • Final editing and completion of group's wiki report.	Grade and give optional general feedback or group specific feedback added to wikis.

	• Submission of wiki report – you should instruct students as to whether you require that the final report be printed and a copy submitted (most wikis allow pages to be saved as pdfs). You may also require that it is submitted in a format to be checked by plagiarism detection software. • An "executive summary" should be included as part of the report. **Individual** • Reflective piece to be submitted. • Laboratory book to be submitted. • Peer assessment of other students in a group (optional).	Grade using guidance notes (see appendix). Grade and add some comments.

Learning Outcomes

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

1. Use a provided literature source to write laboratory procedures to complete absorption spectroscopy, adsorption studies and photocatalytic degradation studies, including a list of materials and equipment required.
2. Prepare a chemical risk assessment for the experimental work to be undertaken.
3. Plan time in the laboratory effectively in order to complete the work assigned.
4. Keep an accurate and current record of experimental details and data in a laboratory notebook.
5. Draw conclusions from experimental data so as to choose the next step in a research process.
6. Produce a professional report, including an executive summary and an assessment of the scope for each step to be improved, that is supported by the relevant experimental data and a laboratory notebook.
7. Prepare an oral presentation on the findings from the study to present to Fercullen Council.
8. Prepare a short individual reflective statement on the group process, transferable skills developed and the extent to which the stated learning outcomes were met.

Transferable Skills

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

- Team work: they work in groups to complete the task assigned, use a wiki to facilitate collaboration and meet between sessions to review progress.
- Organisation and planning: they prepare procedures on a suitable scale and plan their time in the laboratory effectively.
- Communication skills: they present (oral presentation) and report (wiki and final report) on the scientific work performed in keeping with the context.
- Drawing conclusions and recommendations from data: they justify decisions, assumptions and conclusions made with reference to results from other group and supporting literature in order to produce a logical and clearly reasoned scientific report.
- Numeracy: analysis of data obtained from experimental results.
- Professional role and responsibilities: they adopt the role of a professional chemist and are required to make a recommendation based on professional opinion.
- Problem solving: they work in groups to address the brief presented in the context scenario.
- Information technology skills: they use a wiki to collaborate and develop their ability to use word-processing, spreadsheet, presentation, chemical drawing and library database software.
- Metacognition: they 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.

Table 3: Schedule of Workshops and Laboratory Classes

Session (3 hrs)	Activities	Learning Outcomes		
		Concepts	**Laboratory Skills**	**Transferable Skills**
1	Introduction and Project Planning	Water Pollution Beer-Lambert Law	N/A	Team work Organisation and planning Problem solving Use a literature source to write laboratory procedures Drawing conclusions and recommendations from data Numeracy Information technology skills
2	Laboratory 1: Beer Lambert Law	Beer-Lambert Law Quality Control information	Prepare chemical risk assessment Plan laboratory time Solution preparation UV/visible spectroscopy	
3	Laboratory 2: Adsorption I	Surface adsorption Langmuir isotherms	Prepare chemical risk assessment Plan laboratory time Solution preparation UV/visible spectroscopy Adsorption studies	
4	Laboratory 3: Adsorption II			
5	Laboratory 4: Photocatalysis Planning	Advanced oxidation processes (photocatalysis) Kinetics	Prepare chemical risk assessment Solution preparation Reactor design Absorbance spectroscopy Centrifugation	
6	Laboratory 5: Photocatalysis			
7	Feedback Clinic	Produce a professional report	N/A	Team work Organisation and planning Problem solving Drawing conclusions and recommendations from data
8	Presentations	Produce a professional report Prepare an oral presentation Prepare a short individual reflective statement	N/A	Team work Communication skills Professional role and responsibilities Metacognition

Session 1: Introduction to Project and Planning First Laboratory Session

The purpose of this induction session is to:

1. Introduce the case study context and outline the schedule of work, the learning outcomes, and assessment components and criteria.
2. Provide information on the background to the experiments to be used during the project and further reading required.
3. Provide information on the references to be consulted to plan the first stage of laboratory work.
4. Demonstrate how to use a sample wiki (adding and editing pages, uploading files and adding comments).

Planning the Session

The aim of this first session is to familiarise the student with the case study and guide them on to developing the first experimental procedure for Session 2 (Beer Lambert Law).

In advance of the session you should:

- Prepare copies of the Student Guide ready for distribution. If you plan to place the guide online, it may be useful to bring a hard-copy of the first session and the Letter from Fercullen Council (pages 7 – 10 in Student Guide) and if you plan to begin planning the laboratory protocol for Session 2 in this session, students will need to have pages 11 – 14 available to them. You may also decide to hand out the entire guide without the appendices, which can be available online.
- Determine the group size and members of each group. A group size of three students is suitable for this project.
- Prepare a wiki for demonstration in the induction presentation, or request assistance from an institution learning technology officer to demonstrate the wiki to be used during the project. Depending on the wiki being used, you may need to set up individual group wikis in advance and add members of each group to their wiki. This may only be possible after you receive email addresses after the induction presentation.

During the session you should:

- Give an induction to the case study. An induction presentation is available as a PowerPoint file. This takes the student through the introductory parts of the Student Guide: overview of the resource, learning outcomes, transferable skills developed, assessment, and the context of the project.
- Provide an overview of the wiki, highlighting how the information is written to the wiki, how student contribution is logged, and the desired overall structure of the final wiki. Extensive details are provided in the Student Guide Appendix 1 on using a wiki.
- Provide students with information on how they may find resources required for the project, or request assistance from an institution librarian to give an overview of library access to journals and databases.

- Briefly give an overview on guidelines for the final presentation. One important element to flag is that if the presentation is to be based on the wiki (rather than a PowerPoint presentation made from the wiki) it is a common mistake for students to just read from the wiki. Good practice for presentations is provided in Appendix 5.
- Ensure that students are clear on required work to be submitted:
    1. "Interim report" in form of draft wiki to be ready for discussion and feedback in Session 7.
    2. Final report for Council Scientific Officer under headings provided with letter - you should specify if this should be submitted in printed form or if the wiki is sufficient, and whether this should be submitted with or after final presentation.
    3. Oral presentation to be given in front of peers in Session 8.
    4. Reflective piece to be submitted one week after all other work is completed. Guidelines for this are included in the appendix to the Student Guide.
- In order to get going smoothly with Session 2 planning, a "Briefing Document" on UV/visible spectroscopy is provided in the Student Guide. After providing some brief overview details of planning and recording laboratory details, it is advisable to get students to begin in their group to prepare for Session 2 in this first session.
- Read over the tasks students should complete before Session 2 to ensure students are clear about what is required. Remember to give a deadline for which draft experimental protocols should be logged on the wiki so that you have time to check them in advance of the laboratory session.

Planning the Next Session

- Ensure all students have access to the VLE or wiki.
- Check the students' wikis to ensure they have completed some work on their protocol. To save time, group the protocols submitted by students into three categories: ready to go with minor amendments at the start of the laboratory class; some changes needed—refer to Briefing Document; no submission—request group contact you immediately. As these will be generic across all laboratory planning, it may be useful to use a version of the feedback text given in the appendix (Appendix 2: Tutor Guide to using wikis). In providing feedback, the emphasis is on getting students to think about the laboratory protocol, rather than telling them how to do it. There is sufficient time in the laboratory class to iron out any details.
- Submit the technical requirements for the laboratory to the person responsible for preparing the materials—details are given below.
- Plan a mechanism for groups to share their findings from the laboratory class with each other. This may be a whiteboard, a projector with an Excel sheet—both of which assume analysis will be done in class—or a space on the VLE/common wiki for students to log their findings.
- Have a chemical safety assessment statement prepared so that a copy is available in the laboratory (safety information on malachite green is provided in Appendix 4. It is suggested that students are presented with solutions rather than handling the powder, depending on their own laboratory experience to date).

Session 2: The Beer-Lambert Law (Laboratory)

The purpose of this session is to carry out the experimental procedure to produce a Beer-Lambert calibration chart for the quantification of malachite green. It has been found that students take some time to adjust to the freedom of developing and running their own laboratory procedures, even with guidance, so this session is purposefully straight-forward experimentally to allow them adjust to this new experience. Students should be able to develop the protocol by effectively engaging with the briefing document provided in their guide (provided here in Appendix).

Planning this Session

During this session you should:

- Provide students with on-going feedback on their experimental protocols, demonstration of instruments, etc. The main difficulty in this experiment will be finding a suitable concentration so the absorbance at the wavelength of maximum absorbance is less than 2.
- Encourage students to formally record laboratory work in their notebooks as they proceed. Discourage “rough-work”!

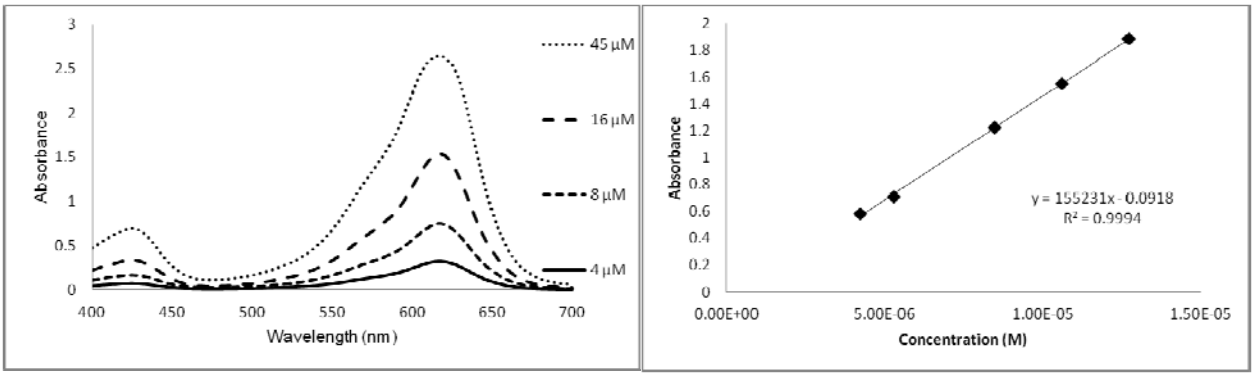
Technical Requirements for this Laboratory Session:

- Per group: 1 x 10^{-3} M stock solution of malachite green solution, 25 ml pipette, 1 ml pipette, 6 - 8 x 50 ml volumetric flasks, distilled water, cuvettes.
- UV/visible spectrometers

Sample Protocol:

1. Prepare a series of appropriate dilutions of the standard solution until the absorbance at the wavelength of maximum absorbance is less than 2. Knowing the maximum possible concentration, prepare five to six solutions with concentrations lower than this value, and measure their absorbance.
2. Continue a series of dilutions until the limit of detection has been reached.
3. Plot absorbance versus solution concentration and determine the extinction coefficient from the slope.
4. Conduct a whole class comparison of the extinction coefficients so as to enable the construction of a QC chart.

Sample Data:



Left: Beer-Lambert absorbance spectra for malachite green oxalate

Right: Beer-Lambert plot to determine the extinction coefficient.

Additional Options:

- You may wish to provide students with a solution whose concentration is not provided so they can test their calibration chart to determine its concentration.

Laboratory notes:

- The extinction coefficient will depend on the counter ion in your malachite green sample.
- You may choose to recycle the solutions from this laboratory in the adsorption and photocatalysis testing laboratory classes.

Planning the Next Session

- Make the paper and supplementary material describing this experiment available to students so that they may plan their experimental protocol. *A Simple Adsorption Experiment*, G. Guirado and J. A. Ayllón, *Journal of Chemical Education*, 2011, **88**, 624–628
- Check the students' wikis to ensure they have completed some work on their protocol. Comment as appropriate.
- Decide on the range of concentrations to be studied by each group (depends on the number of groups in your study). A range of 1-50 $\times$ 10^{-6} M is appropriate. By assigning 3 to 4 different concentrations per group, the class will have a large data set to share.
- Submit the technical requirements for the laboratory to the person responsible for preparing the materials—details are given below.
- Plan a mechanism for groups to share their findings from the laboratory class with each other. This may be a whiteboard, a projector with an Excel sheet—both of which assume analysis will be done in class—or a space on the VLE/common wiki for students to log their findings.
- Have a chemical safety assessment statement prepared so that a copy is available in the laboratory (safety information on malachite green is provided in Appendix 4. It is suggested that students are presented with solutions rather than handling the powder, depending on their own laboratory experience to date).

Session 3: Adsorption Experiments I (Laboratory)

The purpose of this session is to allow students familiarise themselves with surface adsorption experiments so that they may optimise their experimental protocol for the *Adsorption Experiments II Laboratory* using one of a range of adsorbents you make available. For this laboratory experiment, filter paper is used as it is convenient experimentally and allows students concentrate on the preparation of solutions and analysis of data.

Planning this Session

During this session you should:

- Provide students with on-going feedback on their experimental protocols, demonstration of instrumentation, etc. Students should draw from their work in Session 2 in suggesting concentration ranges to study. The main difficulty in this experiment will be ensuring the mass of filter paper is consistent across the groups.
- Encourage students to formally record laboratory work in their notebooks as they proceed. Discourage "rough-work"!
- Set deadline for completion of work on wiki before next session.

Technical Requirements for this Laboratory Session:

- Per group: 6 x sample containers (20 ml capacity), adsorbent material (filter paper), standard solution of malachite green, cuvettes.
- UV/visible spectrometers

Sample Protocol: (see Guirado and Ayllon, J. Chem. Ed., 2011, 88, 624-628 for extensive details)

1. Prepare a stock solution of malachite green oxalate solution ($1x10^{-4}$ M)
2. Allocate groups of students to test the absorbance of the filter paper.
3. Place 20 ml of known conc. of malachite green into a sample container.
4. Note the absorbance of each container prior to addition of the filter paper.
5. Place the filter paper into each container and stopper.
6. Take a volume for UV/Vis analysis every 30 min and return to the beaker when finished.
7. Plot a graph of concentration vs. time and comment on the data observed.
8. Compare the groups' findings.
9. Investigate the Langmuir isotherm information for each material. A detailed description of this is provided in the Guirado and Ayllon paper and supplementary information.

About Langmuir Isotherms

The adsorption of a molecule onto a solid surface can be modelled by surface adsorption isotherms. The two most common are Langmuir and Freundlich isotherms. For a coloured solution, if an adsorbent such as filter paper is added, as the molecules deposit onto the surface, the colour of the solution will become less intense. This is conveniently measured by absorption spectroscopy.

The Langmuir isotherm relates the ratio of mass of adsorbent (*w*) and amount of dye adsorbed onto the surface (n_s) to the equilibrium concentration C_{eq}. From its linear form:

$$\frac{w}{n_s} = \frac{w}{n_{\max}} + \left(\frac{w}{n_{\max} b}\right)\frac{1}{C_{eq}}$$

a plot of w/n_s vs $1/C_{eq}$ will be linear, with slope $w/n_{max}b$ and intercept w/n_{max}. Therefore, the amount of molecules that can form a monolayer on the surface (i.e. a measure of the capacity of the surface to adsorb) is provided by n_{max}.

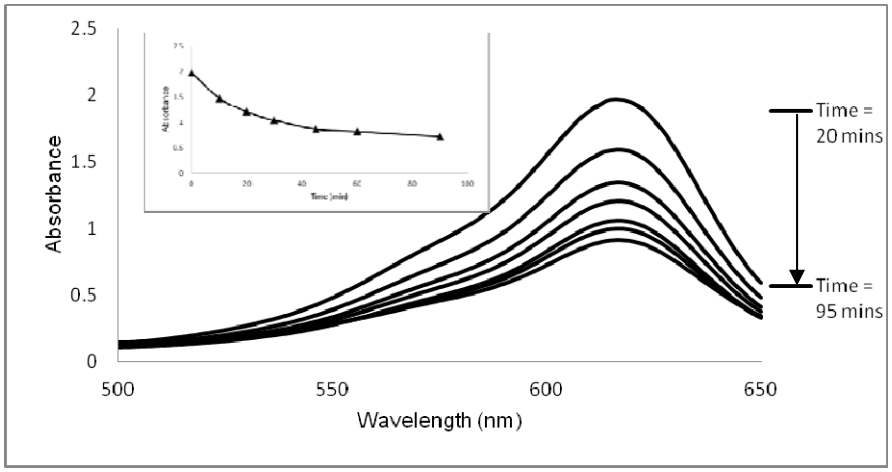
From the experimental data, students can easily determine *w*, the mass of the adsorbent used. The equilibrium concentration C_{eq} is determined from the absorbance of the solution, using the extinction coefficient determined in the previous session. To determine n_s, the calculation involves the use of the mass-balance relationship:

$$n_s = (C_0 - C_{eq}) \times V \times M_w$$

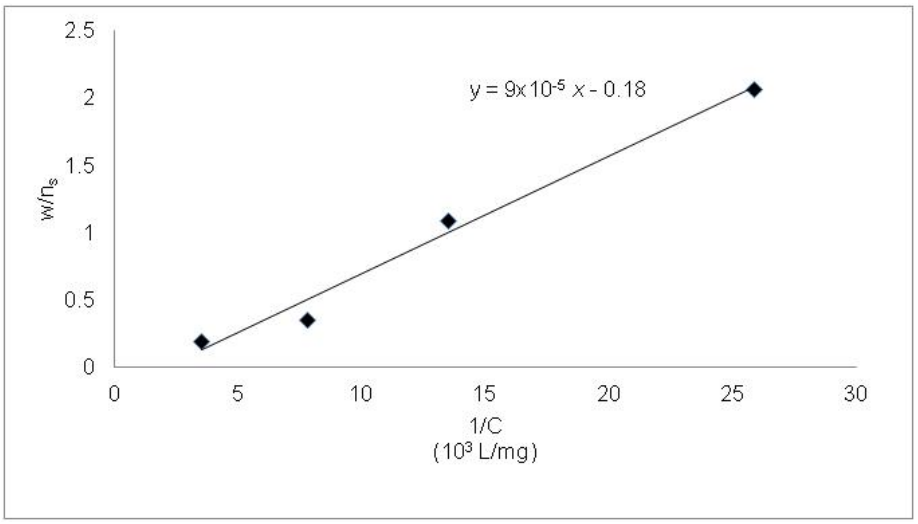
In order to obtain a true value for C_{eq}, it may be necessary to re-measure the absorbance after one week. This can be investigated by students to see if it significantly affects the results.

The capacity for adsorption can also be monitored simply by looking at the concentration dependence over time. Students measure the absorbance at intervals over the laboratory session and compare (if different adsorbents are used by different groups) how quickly the absorption occurs. Therefore depending on the level students are at when completing this resource, you can opt to determine the effectiveness of an adsorbent by considering how much material is adsorbed after a particular amount of time or complete a Langmuir analysis to evaluate n_{max}. Langmuir isotherms assume monolayer coverage.

Sample Data:



Absorbance vs. wavelength over a time period for a piece of filter paper placed in a 1.35 x10^{-5} M solution of malachite green oxalate.(Inset: profile of absorbance at wavelength of maximum absorbance over time).



Graph of adsorbent to adsorbed ratio vs. inverse of equilibrium concentration for 1 piece of filter paper with a mass w = 352 mg placed in an array of malachite green oxalate solutions. n_s = mass of analyte adsorbed onto the adsorbent per litre, C = equilibrium concentration of analyte remaining in solution (units mg/L or mol/L will determine units of b, a constant). From this data, the maximum adsorption n_{max} can be obtained from the intercept – in this case n_{max} = 352mg/0.18 = 1.96g

Additional Options:

- A key factor here is the mass of the adsorbent – you may wish to have different groups investigate different masses so that a comparison can be made.
- Adsorption depends on temperature. Temperature variation allows for thermodynamic information to be calculated.

Laboratory notes:

- In calculation for the Langmuir isotherm, the equilibrium concentration is required. If it is possible, re-measure the absorbance in the following laboratory practical session to see if this value changes from that determined at the end of this practical session.

Planning the Next Session

- Determine the adsorbents you wish to use and assign them to groups. A substantial number are detailed in *Adsorption of methylene blue on low-cost adsorbents: A review*, M. Rafatullaha, O. Sulaimana, R. Hashima, A. Ahmadb, *Journal of Hazardous Materials*, 2010, **177,** 70–80.
- Submit the technical requirements for the laboratory to the person responsible for preparing the materials—details are given below.
- Check the students' wikis to ensure they have completed some work on their protocol. Some comments on providing feedback are detailed in Session 2 (Beer-Lambert experiment).
- Decide on the range of concentrations to be studied by each group (depends on the number of groups in your study). A range of 1-50 $\times$ 10^{-6} M is appropriate.
- Plan a mechanism for groups to share their findings from the laboratory class with each other.
- Have a chemical safety assessment statement prepared so that a copy is available in the laboratory (safety information on malachite green and titanium dioxide is provided in Appendix 4. It is suggested that students are presented with solutions rather than handling the powder, depending on their own laboratory experience to date).

Session 4: Adsorption Experiments II (Laboratory)

The purpose of this laboratory session is to study the adsorption of malachite green onto an adsorbent material. The practical aspect builds on the previous session's laboratory session, where students developed the protocol using filter paper. This laboratory offers significant scope in implementation in terms of the variety of conditions that can be considered. There are several model adsorbents that can be tried. Activated charcoal and silica are typical adsorbents used in surface chemistry laboratory procedures. In this resource, titanium dioxide was examined (sample results below) as it leads into the photocatalysis experiments in the following session. However, there is a growing trend to use low cost natural materials (see review cited in Student Guide: *J. Haz. Mat.,* 2010, **177**, 70 – 80). Depending on capacity to source these materials, there is a lot of scope on the type of adsorbents that could be trialled across a class group.

Planning this Session

During this session you should:

- Provide students with on-going feedback on their experimental protocols, demonstration of instrumentation, etc.
- Encourage students to formally record laboratory work in their notebooks as they proceed. Discourage "rough-work"!

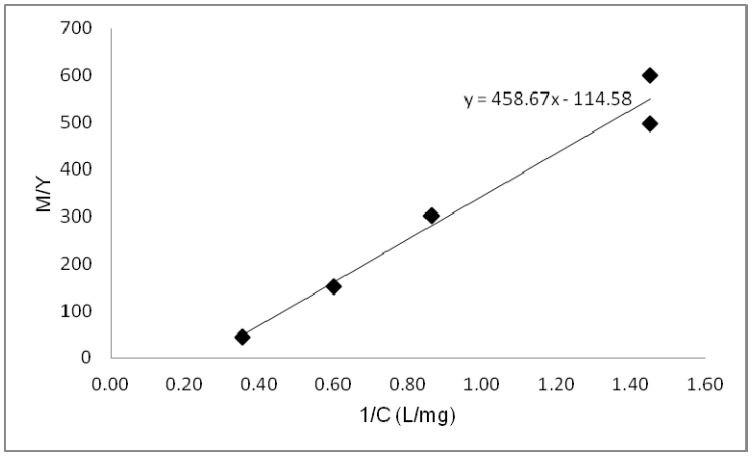
Technical Requirements for this Laboratory Session:

- Per group: 6 x sample containers (20 ml capacity), adsorbent material (to be decided, ~0.5-1g per group), standard solution of malachite green, cuvettes.
- UV/visible spectrometers

Sample Protocol:

Similar to Adsorption Experiments I (Session 3).

Sample Data:



Langmuir isotherm for TiO_2 (Degussa P25) added to various concentration of malachite green oxalate

Additional Options:

- As mentioned above, several different adsorbents are possible, including activated carbon, zeolites, clays, baker's yeast, grass waste.

Laboratory notes:

- In calculation for the Langmuir isotherm, the equilibrium concentration is required. If it is possible, re-measure the absorbance in the following laboratory practical session to see if this value changes from that determined at the end of this practical session.

Planning the Next Session

- Determine which parameters you wish to investigate. Mass of photocatalyst is the parameter most easily changed. You may also have several light sources which would allow for variation in wavelength of light to be studied.
- Check the students' wikis to ensure they have completed some work on their protocol. Some comments on providing feedback are detailed in Session 2 (Beer-Lambert experiment).
- Plan a mechanism for groups to share their findings from the laboratory class with each other. This may be a whiteboard, a projector with an Excel sheet—both of which assume analysis will be done in class—or a space on the VLE/common wiki for students to log their findings.
- Have a chemical safety assessment statement prepared so that a copy is available in the laboratory (safety information on malachite green and titanium dioxide is provided in Appendix 4. It is suggested that students are presented with solutions rather than handling the powder, depending on their own laboratory experience to date).

Session 5: Photocatalysis Experiments I (Laboratory)

The aim of this session is to optimise the protocol for photocatalysis experiments. Several parameters can be varied—most commonly the optimisation of mass of catalyst, the pH of the solution, and the light source. By allowing different groups to attempt different conditions, an optimum set of parameters can be developed over the course of this session, in preparation for the following session. This requires good advance planning and good communication between groups after the laboratory class.

Different brands of TiO_2 show different effectiveness. The industrial standard is "Degussa P25" which is active for many reasons, including its small particle size and the combination of two crystal forms of TiO_2 (anatase and rutile). Millennium chemicals also produce an active form of TiO_2.

The experiment is most commonly carried out using a known mass of powder and irradiating with a light source, which for safety reasons is usually a visible light source (e.g. light bulb). This will involve centrifuging the solution before a sample can be taken for UV/visible absorption analysis. An alternative to this is to adsorb the TiO_2 onto a surface (e.g. silica on glass, filter paper) which will reduce the photocatalytic activity but make the analysis step easier. The data shown below is for the powder/centrifuge method.

TiO_2 does not absorb much visible light, and the mechanism of degradation is therefore thought to be via sensitisation. Some details are provided below.

Planning the Session

During the session:

- This session is about experimentation, so once the initial parameters are agreed (mass of catalyst, light source, etc.) allow the students to experiment with sampling times. While in the next session they may decide to centrifuge all samples at once at the end, this session they will likely have to centrifuge at intervals to see if any degradation is observed at the time intervals selected. As a guide, measurement intervals of ~20–30 minutes are appropriate with an efficient catalyst, a visible light source, and a loading of ~60 mg of TiO_2 in 50 mL (10^{-5} M) solution.
- Because of the nature of this session, students' recording of laboratory details will begin to indicate whether they have been properly maintaining in-laboratory notes over the last few sessions. This is a good time to emphasise the research nature of the work and how maintaining a clear laboratory notebook keeping is part of that.

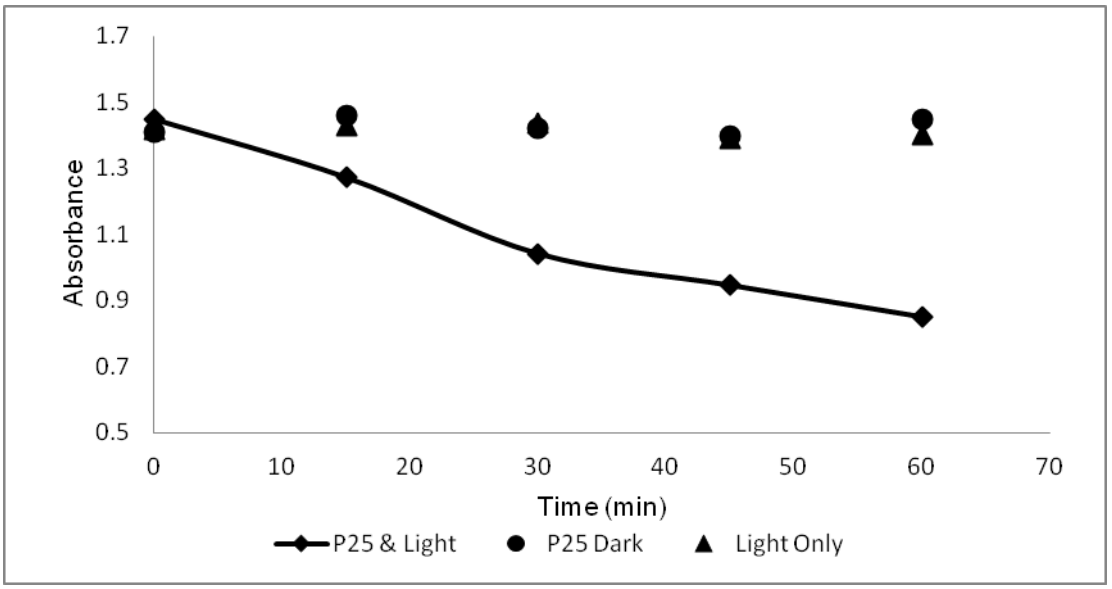
Technical Requirements for this Laboratory Session:

- Per group: 1 x stopwatch, 1 x 400 ml beaker, 1 x 250 ml beaker, 2 large elastic bands, 1 x j-cloth, 1 retort stand, 1 x clamp, 1 x magnetic stirrer & pellet, 1 x low wattage fluorescent bulb ~11W or a 60W incandescent bulb, 1 x power lead & bulb connection, 6 x solution sample containers suitable for use in a centrifuge, 6 cuvettes, various brands of TiO_2.
- UV/visible spectrometer, centrifuge.

Sample Protocol:

1. Set up a photoreactor as per instructions. This can be as simple as suspending the bulb over a beaker containing the solution and TiO_2. A reactor made of two beakers (one inside the other) can also be used so as to protect the bulb from splashes, although this may reduce the light intensity.
2. Dilute stock malachite green to an appropriate concentration (~10^{-5} M) and volume (>50 mL).
3. Measure the appropriate mass of TiO_2 (for the conditions in (2), a range of 50 - 200 mg is appropriate to study).
4. Mix the TiO_2 with the malachite solution for approx 15 mins in the dark. Sampling before and after addition will demonstrate that light is necessary for the degradation of malachite green.
5. Irradiate the samples continuously and withdraw aliquots for analysis. Centrifuge for ~15 mins at 4000 rpm. Alternatively, when a full set of aliquots are ready, number will depend on the model of centrifuge, process all together at 4000rpm for 15mins.

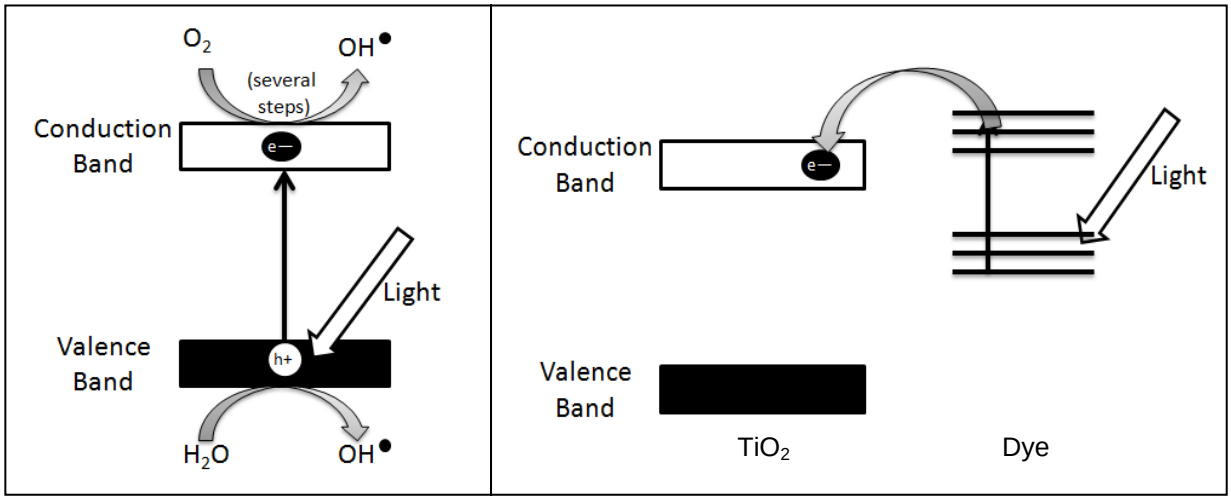
Sample Data:



Graph of absorbance vs time for malachite green oxalate containing Degussa P25 after irradiation with 11 W CFL bulb. Also shown are the absorbance of the solution without irradiation and without TiO_2, to verify the role of both catalyst and light in degradation.

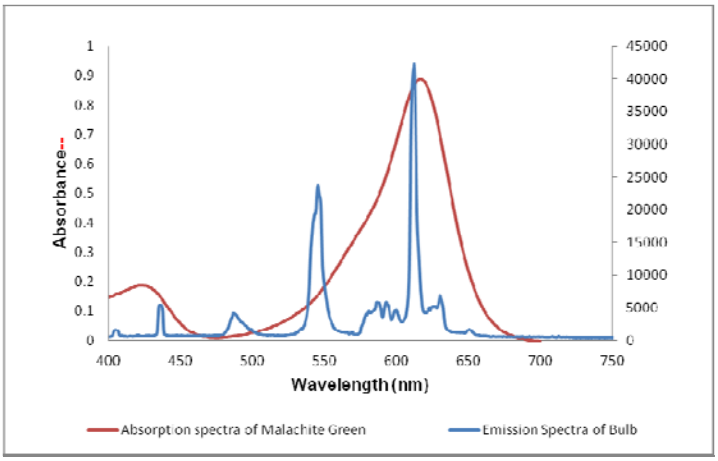
Photocatalytic Degradation of Malachite Green

Semiconductors such as titanium dioxide act as photocatalysts—they become catalytic agents on absorption of light at an appropriate wavelength. The process is illustrated in the diagram below. Light absorption results in the promotion of a valence band electron to the conduction band, leaving a hole in the valence band. Holes are powerful oxidisers, and will quickly oxidise any water present at the surface to hydroxyl radicals. The electron, promoted to the conduction band, is a strongly reducing species, and will oxidise oxygen present at the surface to superoxide anions, which ultimately leads to hydroxyl radical formation as described above. These hydroxyl radicals will lead to the decomposition of organic matter.



Photocatalysis of a dye after light absorption (left) or sensitisation (right)

A problem with titanium dioxide is that is only absorbs a small amount of visible light, and therefore the amount of electron-hole separation that occurs when visible light is used is small. A second mechanism may also occur, whereby the titanium dioxide conduction band is populated with an excited state electron from the malachite green, effectively resulting in oxidative degradation of the dye. This process is known as degradation by sensitisation.



A comparison of the emission spectrum of the photo reactor bulb (11W Philips Genie (CFL)) with the absorption spectra of the malachite green solution.

Planning the Next Session

- Check the students' wikis to ensure they have completed some work on their protocol. Some comments on providing feedback are detailed in Session 2 (Beer-Lambert experiment).
- Plan a mechanism for groups to share their findings from the laboratory class with each other. This may be a whiteboard, a projector with an Excel sheet—both of which assume analysis will be done in class—or a space on the VLE/common wiki for students to log their findings.
- Have a chemical safety assessment statement prepared so that a copy is available in the laboratory (safety information on malachite green and titanium dioxide is provided in Appendix 4. It is suggested that students are presented with solutions rather than handling the powder, depending on their own laboratory experience to date).

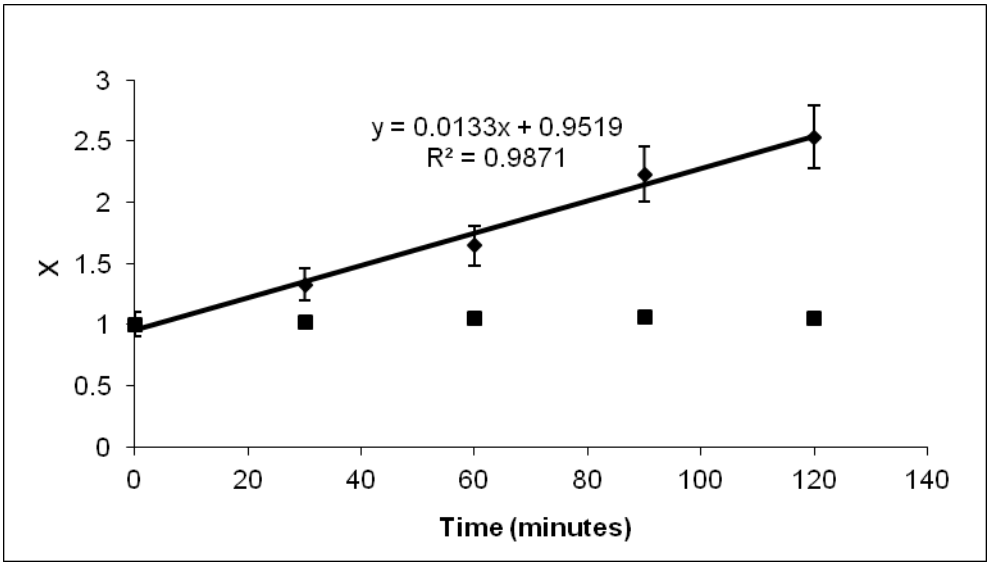
Session 6: Photocatalysis Experiments II (Laboratory)

The aim of this session is to allow students repeat their photocatalysis experiments based on the optimised protocol from the previous session. It will also provide some opportunities for students to discuss their final report requirements.

Planning this Session

Experimentally, this session is similar to the last one (Session 5). The additional analysis required is for students to quantify the degradation using kinetic analysis on their absorbance data. This is most commonly a first order analysis, obtained by plotting the natural log of the ratio of absorbance to initial absorbance against sample time.

An example of a kinetic analysis of degradation is shown below (*Chem. Educ*. 2006, **11**, 184-186). In this analysis, $\ln(A_0/A_t)$ is plotted as a function of time t. This is a first order rate plot, which if linear produces the rate constant from the slope of the graph. This will allow students to compare the degradation parameters to see which set is most effective.



Plot of $\ln(A_0/A_t)$ [=X] versus t (circles) with best-fit slope. Squares show data for samples that were not irradiated.

Additional Options

- One of the limitations of photocatalysis as presented here is that TiO_2 is mainly activated by UV. The synthesis of N-doped TiO_2 is facile, and generates a visible light active material. This synthesis and full explanatory notes are available in the "*Small Materials to Solve Big Problems*" C/PBL resource.
- The rate of degradation depends on pH and the presence of oxygen. Variation in experiments could be included by examining a broad pH range across the group.

Session 7: Feedback Clinic

Aim of this Session:

The purpose of this workshop session is to:

- Answer any student queries on the assignment and activities, and discuss any issues raised.
- Provide learners with formative feedback (as an entire class and in their groups) on which areas of the report they need to work on, and which ones have been addressed satisfactorily.
- Identify the tasks remaining, and plan for when they'll be achieved.

Running this Session

In this workshop, students have the opportunity to receive formal feedback from their tutor who will highlight any areas that require work, address any misunderstandings and identify the strengths of the work produced to date. It also gives them the opportunity to review their work as a group, and to complete any tasks they have not been able to do to date while you are speaking to other groups.

It is probably best to speak to the entire cohort first and deal with any general issues, and then speak to each group individually in turn; this may be best done with the help of an additional tutor to ensure each group gets adequate attention. It may be worth reminding students to take some notes.

Some specific guidelines for the final presentation that will take place in Session 8 (Presentations) should be provided. It is worth reminding students that presentations should not take the form of "reading the wiki". General guidelines for preparing presentations are provided in the appendices to the *Student Guide*. You will need to indicate to the students whether peer evaluation will take place, how long the presentation should last and whether it should be in a PowerPoint format or presented directly from the wiki pages. If there are a lot of groups, you may decide to ask each group to focus on one particular section of the report. Students should be reminded to refer back to the original brief from Fercullen Council to ensure that their presentation and report are addressing all aspects.

During the time you are speaking to an individual group, the other ones will be able to plan the work required to produce and give their presentation and any other work that still remains.

Session 8: Oral Presentations

The purpose of this workshop session is to:

- Have students present their group's work to their peers and tutor by summarising the work undertaken and their recommendations and to answer any questions put to them.
- Provide oral feedback to students (with optional written feedback on each presentation from tutor and peers)
- Support students learning about alternative approaches to the project from the other presentations, while providing a supportive audience and constructive feedback to their peers.
- Remind students of final deadlines for completion of work.

In this workshop, each group will present their work and their recommendations to Fercullen Council, and will listen to and assess the presentations of the other groups. You may opt to invite some guest tutors who have a background in physical chemistry, or who work or have worked in this sector 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. 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. It is recommended that the feedback form at the end of this section be used to give some written feedback to each group member, and that some strengths should be noted as well as areas for improvement. The forms can be printed two to a page.

If each student is given a form and asked to write their name on the back of it at the beginning of the session, they can then swap them with another group so that each student will be providing feedback to one other person. The system can be kept anonymous if the tutor distributes the forms initially, and then collects them at the end and hands them back out to the person whose name is on the back.

Students should be reminded about the deadline for the submission of their final proposal (generated from the wiki by printing it directly or converting to PDFs and then printing, depending on the software). It is expected that any feedback (oral or written) relevant to the report provided by the tutor after the presentation will be incorporated. Indicate whether you want them to have submitted the report through a plagiarism detection system prior to submission.

Students should also be reminded about the deadline for submission of their reflective piece which is usually be several days after that for the group report. Guidelines for the reflective piece are given in the Student Guide appendices. If you have opted to include peer assessment by each group member, they should be asked to complete a form on which they rate the frequency and quality of contributions of each other member to the group.

PRESENTATION SKILLS EVALUATION FORM

Speaker/ Group:

Topic:

What I liked:

Suggestions:

Date **Signed (Optional):**

PRESENTATION SKILLS EVALUATION FORM

Speaker/ Group:

Topic:

What I liked:

Suggestions:

Date **Signed (Optional):**

Appendices: Tutor Guidelines

The following guidelines are given to help you in preparing your laboratory work and reporting requirements:

1. Student Briefing Pack
2. Tutor Notes on Using Wikis
3. Technical Requirements for Laboratory Sessions
4. Chemical Safety Information.

Appendix 1: Student Briefing Pack

Fercullen District Council

Office of Environmental Control and Remediation

FDC

Foton House, County Offices, Fercullen.

To: Whitewater Environmental Services

Re: Analysis and remediation of polluted water at Fercullen Fish Farm

As you may have seen in local media reports, a recent accident at a local fish farm resulted in a spillage of a large quantity of malachite green into the main holding tank at the farm. Malachite green is not permitted for use in aquaculture and was being used illegally. As a result, the owner was reluctant to alert the authorities of the spillage, and did not do so until our monitoring stations detected some small amounts of the substance downstream in another licensed fishing area.

We wish to request your services in dealing with this problem. Having had a consultation with our Scientific Officer, we would like the following work to be carried out:

- Analysis of the concentration of malachite green.

- Determination of the effectiveness of removal of this pollution source from the spill site by developing and trialling protocols for remediation of water. Our Scientific Officer has determined two techniques from the Royal Society of Chemistry *Sustainable Water document* that may be suitable:
    - Adsorption onto low-cost adsorbents.
    - Metal oxide photocatalysis.

- Compile a report for our Scientific Officer recommending an appropriate strategy for dealing with this spillage in order to prevent the consequent pollution of nearby fishing areas. Justification of this recommendation is required, along with costing of the adsorbent. An interim report should be submitted to our Scientific Officer by an agreed date.
- Prepare an executive summary that can be used as a basis for a press-release for local media outlining the extent of the problem and how the spillage will be dealt with effectively.
- In order to fulfil subsequent Freedom of Information requests easily, all work completed for project, including data collected, analysis and conclusions drawn should be logged on a website – a wiki is most appropriate.

You will report directly to our Scientific Officer. The house guidelines for structuring a report for Fercullen District Council are given overleaf. We look forward to working with you in dealing with this issue in as rapid and effective a manner possible.

Yours sincerely,

Emer Power

E. Power

Fercullen District Council

Office of Environmental Control and Remediation

FDC

Foton House, County Offices, Fercullen.

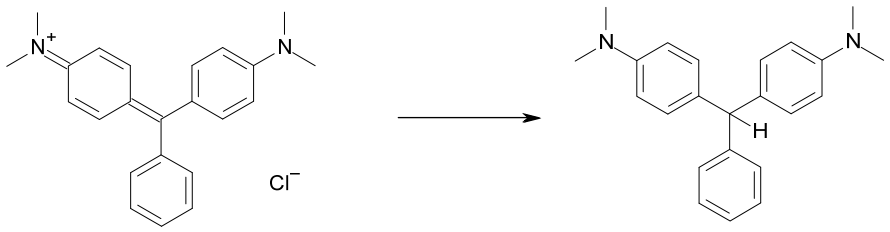
Guidelines for Structure of Scientific Reports for Fercullen District Council

Reports should be a maximum of 2,000 words (not including figures, tables etc.) and should contain the following elements:

1. Report title, date submitted and author name(s).
2. An executive summary (aimed at a non-technical audience, maximum 200 words)
3. The main body of the report to include:
    - Aims and objectives
    - Background information and related previous work
    - Experimental details
    - Results and discussion
    - Conclusion including recommendations
    - Glossary of definitions for all terminology used to ensure clarity.
4. References should be formatted according to the Royal Society of Chemistry Publishing author guidelines format. (See page eleven of the document at this link: http://www.rsc.org/images/Guidelines_tcm18-186308.pdf)

🕮Briefing Document: UV/Visible Spectroscopy

Malachite green is a triphenylmethane salt (Figure 1) which has as intense green colour. It had been a popular anti-fungal agent in fish farming, but after concerns about the toxicity of the material it was banned in many countries for this purpose. However, it is still used illegally because it is effective and cheap. Fish in water exposed to malachite green have been found to contain high amounts of leucomalachite, the reduced form of malachite green. The intense colour of malachite green is easily detected by UV-visible absorption spectroscopy.



Malachite green

Leuco malachite green

Figure 1: Malachite green and leucomalachite green

UV/VIS Spectroscopy is used to measure the **absorbance** of a solution. Absorbance (A) is defined as the logarithm of the ratio of the intensity of light (radiation) striking the sample (I_0) at any wavelength divided by the intensity of light transmitted through the sample. Assuming no other physico-chemical factors are involved (e.g. aggregation), the measured absorbance is proportional to the concentration of a substance in solution (C) for a given path-length (*l*). The constant of proportionality is known as the extinction coefficient (ε). This relationship is expressed in the Beer-Lambert Law:

$$A = \varepsilon.C.l$$

If concentration has units mol.dm^{-3} and the cell path-length is 1 cm, then the units of ε are dm^3 mol^{-1} cm^{-1}. In a typical experiment to analyse the Beer-Lambert law, a range of solution concentrations are prepared and the absorbance at the wavelength of maximum absorbance is measured (λ_{max}) for different concentrations, using solvent as a reference. A plot of the graph A against C will therefore produce a linear calibration curve, with slope ε. Because of the definition of A (ratio of incident light over ratio of transmitted light), absorbance values of less than 2 are considered suitable.

The following details are usually recorded for a UV/visible spectroscopy experiment. Use this information to help you plan the experiment you carry out.

- Identity of the substance (name, date of analysis, nature of substance (solid or liquid), structural formula
- Test conditions (spectrometer (name, type, operational parameters (e.g. slit width)), cell type (e.g. silica quartz, glass, PMMA), wavelength range (e.g. 350 – 700 nm), path length (e.g. 0.1 –10 cm), solvent (e.g. water, methanol, acetonitrile), concentration of test substance, test temperature (e.g. 20-25 ºC), pH environments used: neutral (pH = 7), acid (pH < 2) and alkaline (pH > 10)

Quality Control

You may wish to extend the determination of your extinction coefficient to placing it in the context of how it compares to other groups who have done the same determination. In this case, each group can complete a quality control chart by calculating the average and standard-deviation of the extinction coefficients.

A QC chart can be constructed by plotting the extinction coefficient each group obtains as shown in Figure 2. It is usual to add in lines to represent the average, warning (2σ) and signal (3σ) lines. The QC chart can then be used to test the accuracy of a subsequent extinction coefficient measurement, which should fall in between the average ± 2σ or average ± 2σ range.

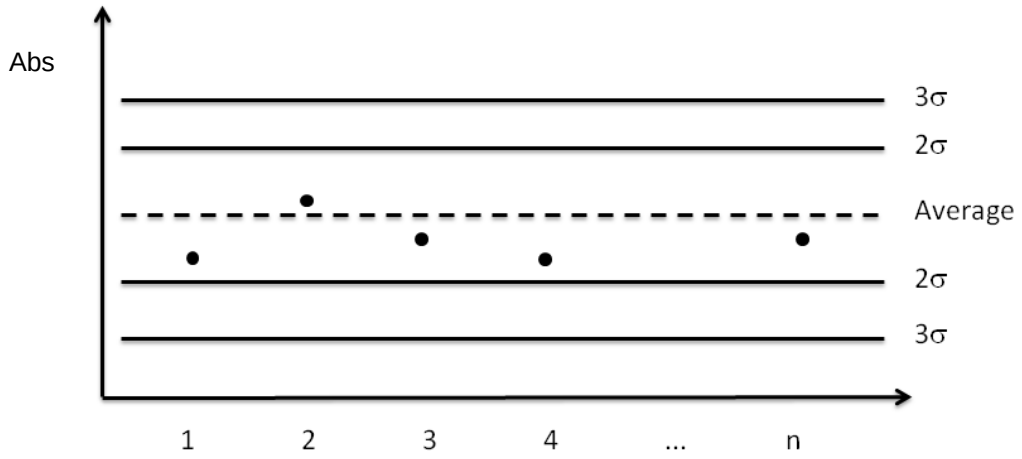


Figure 2: A sample quality-control chart showing average of data, 2σ, and 3σ lines

Limit of Detection and Limit of Quantitation

It is also possible to determine the limit of detection (LoD) – the lowest concentration that an analyte in a sample that can be detected (not quantified) – and the limit of quantitation (LoQ) – the lowest concentration that an analyte in a sample can be quantified. These are conveniently determined by successive dilutions until a signal to noise ratio of 10 is reached for LoQ and 3 is reached for LoD. These values will illustrate the suitability of UV/visible spectroscopy for analysing the presence of small amounts of malachite green in the sample.

The values obtained from the QC analysis should be included in the UV/visible results for the first laboratory session.

Literature References Provided to Students

Session 2:

A Simple Adsorption Experiment, G. Guirado and J. A. Ayllón, *Journal of Chemical Education*, 2011, **88**, 624–628 (*see also supplementary information*)

Session 3:

Adsorption of methylene blue on low-cost adsorbents: A review, M. Rafatullaha, O. Sulaimana, R. Hashima, A. Ahmadb, *Journal of Hazardous Materials*, 2010, **177,** 70–80.

Session 4:

Photodegradation of methylene blue: Using solar light and semiconductor (TiO_2), R. F. P. Nogueira and W. F. Jardim, Journal of Chemical Education, 1993, **70 (10)**, p 861-862.

Photodegradation of malachite green under natural sunlight irradiation: Kinetic and toxicity of the transformation products, L. A. Pérez-Estrada, A. Agüera, M. D. Hernando, S. Malato, A. R. Fernández-Alba, Chemosphere, 2008, **70(11)**, 2068-2075.

Session 5:

The Use of Titanium Dioxide-Mediated Photocatalysis in the Treatment of Wastewater: An Undergraduate Laboratory Experiment, MK Seery, L Clarke, SC Pillai, Chem. Educator, 2006, 11, 1-3.

Appendix 2: Tutor notes on using wikis

A wiki is "a collaborative website consisting of one or more pages that allow authorised users to contribute to or edit page content." (source: http://usermanual.pbworks.com/Glossary)

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 pdfs). 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 end of this section). Access to wikis is controlled by the administrator providing a safe online working environment.

An existing wiki on environmental chemistry is available here:
https://sites.google.com/site/environmentalchemistryoptione/home

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 https://plans.pbworks.com/academic to sign up and http://usermanual.pbworks.com/w/page/11632089/Home for help)

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 laboratory 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.

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 that the groups 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. Each group is required to post a short summary of their weekly meeting (decisions made and resulting actions, people responsible and dates to be completed) and, in this way, work undertaken that may not otherwise be apparent from the wiki is captured.

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://www.youtube.com/watch?v=gRj5ABJ-IPY&feature=related).

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://online.cofa.unsw.edu.au/sites/default/files/episode-pdf/CS_Wikis_LTTO.pdf

Further Reading / Viewing:

A video showing how to set up a PBworks wiki for educational use:
http://www.youtube.com/watch?v=SZ5OV14v4xU

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 https://blog.itu.dk/MVOL-F2010/files/2010/02/corporate-wiki-users-results-of-a-survey.pdf
- A wiki to develop policy in the area of green chemistry in California is available here: http://cagreenchem.wikidot.com/start and here: http://cagreenchem.wikidot.com/welcome ; http://eponline.com/articles/2009/02/09/calif-launches-wiki-to-develop-green-chemistry-regulations.aspx
- Ganfyd wiki is a medical database that can be edited by registered medical practitioners and viewed by anyone: http://www.ganfyd.org/index.php?title=Main_Page
- 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:

- Chemistry Education Research and Practice paper that discusses using 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.
- J Chem Ed article on using wikis to promote collaboration on online laboratory 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 Environmental Chemistry 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 post some minutes with a summary of group decisions as soon as possible. It is recommended to assign two roles in meetings that rotate each week - a "chair" (organises meetings, decides on agenda and running order, and deals with any differences of opinion), and a "recorder" (records a summary of the decisions made and resulting actions, people responsible, and dates to be completed meetings). 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

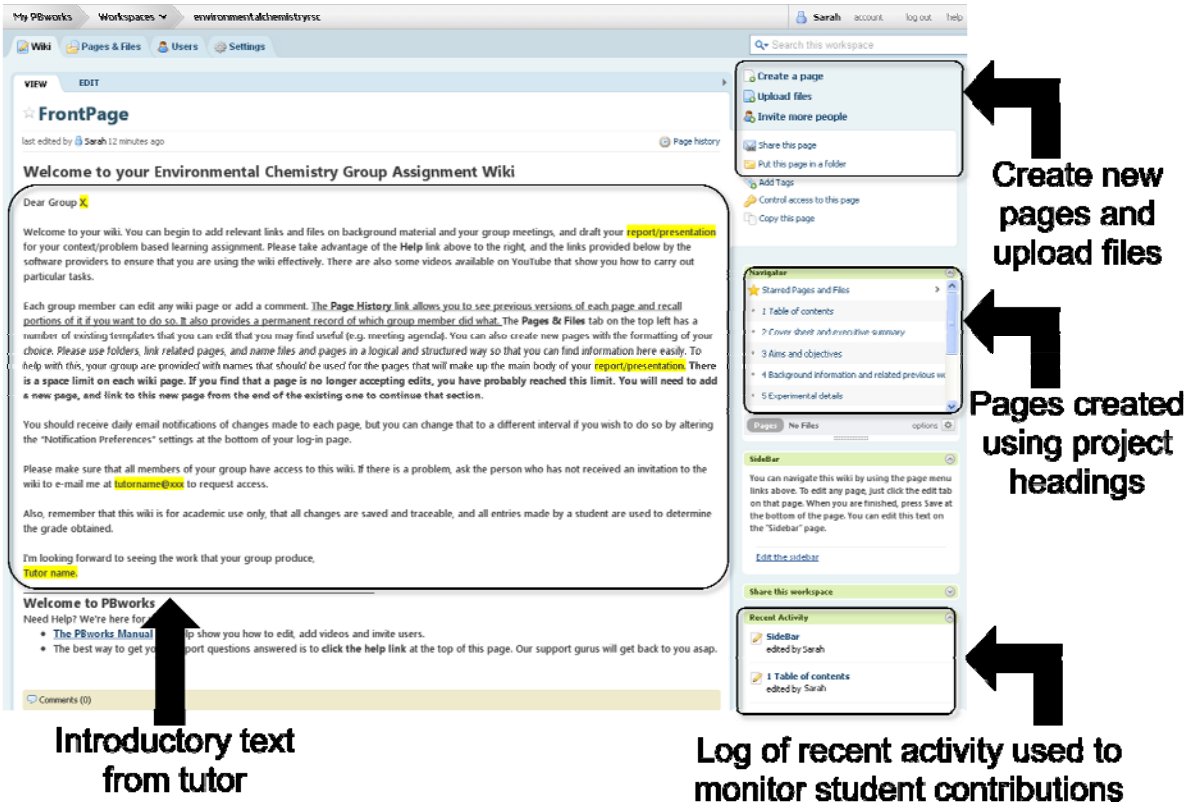
As far as possible, any comment on an improvement that could be made was prefaced by a positive comment on another aspect of the wiki.

- 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.

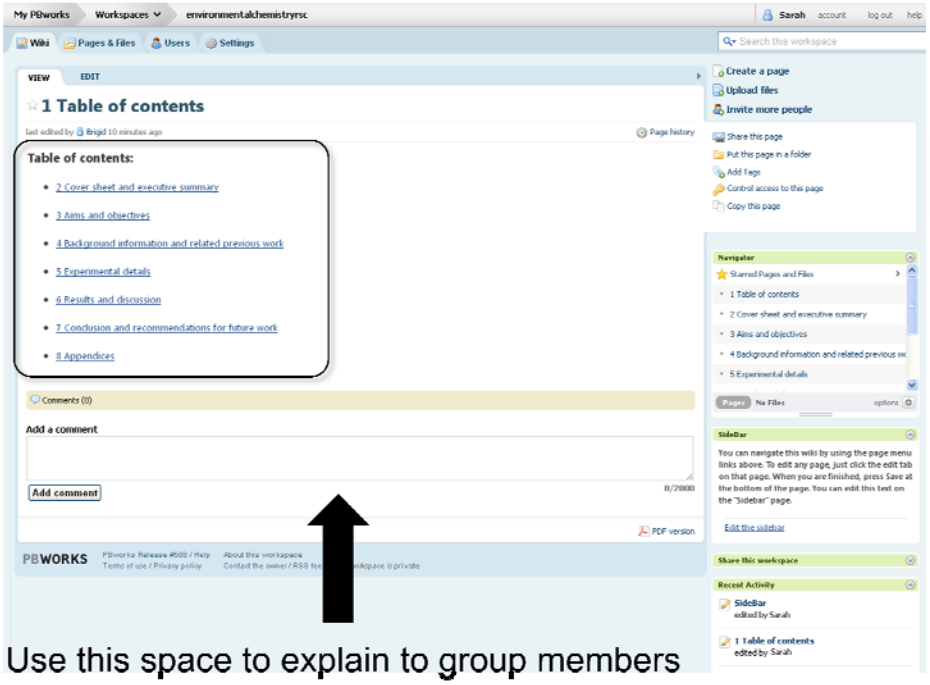
General Feedback and Technical Support Comments

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

- Some of you put a lot of work into your procedures (well done) and some did not do as much. Please note that any feedback you get should be acted on by making changes to your second step procedure for next week (e.g. provide a list of glassware and equipment needed and the reaction scheme showing the starting material and product structure). Also, the final report should have the corrected version of these procedures and the safety information, and the original ones I corrected should be handed back then also.
- Most of you made a good attempt at how you will analyse the data. Some comments still left to address are....
- 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.



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 3: Technical Requirements for Laboratory Sessions

Session 2 (Laboratory 1) – The Beer-Lambert Law

- Per group: 1 x 10^{-3} M stock solution of malachite green solution, 25 ml pipette, 1 ml pipette, 6 - 8 x 50 ml volumetric flasks, distilled water, spectrometer, stock of cuvettes & Pasteur pipettes
- UV/visible spectrometers

Session 3 (Laboratory 2) – Adsorption Experiment I

- Per group: 6 x sample containers (20 ml capacity), adsorbent material (filter paper), standard solution of malachite green, UV/vis spectrometer, cuvettes,
- UV/visible spectrometers

Session 4 (Laboratory 3) – Adsorption Experiment II

- Per group: 6 x sample containers (20 ml capacity), adsorbent material (to be decided), standard solution of malachite green, UV/vis spectrometer, cuvettes,
- UV/visible spectrometers

Session 5 & 6 (Laboratory 4 & 5) – Photocatalysis I and Photocatalysis II

- Per group: 1 x stopwatch, 1 x 400 ml beaker, 1 x 250 ml beaker, 2 large elastic bands, 1 x j-cloth, 1 retort stand, 1 x clamp, 1 x magnetic stirrer & pellet, 1 x low wattage fluorescent bulb ~11W or a 60W incandescent bulb, 1 x power lead & bulb connection, 6x solution sample containers suitable for use in a centrifuge, 6 cuvettes, analytical mass scales, various brands of TiO_2.
- UV/visible spectrometers

Appendix 4: Chemical Safety Information

The following information is for malachite green (solid form). It is suggested that solutions are prepared and presented to students as dilute solutions (< 10^{-3} M).

Substance name	TiO_2	Malachite Green
CAS no	134-63-67	633-03-4
Conc	solid	solid
Amount used	<1g	<1g
Hazard Classification	Not a hazardous substance or mixture according to Regulation (EC) No. 1272/2008.	Reproductive toxicity (Category 2) Serious eye damage (Category 1) Acute aquatic toxicity (Category 1) Chronic aquatic toxicity (Category 1) Acute toxicity, Oral (Category 4)
Hazard statement/Risk phrase	This substance is not classified as dangerous according to Directive 67/548/EEC.	H302 Harmful if swallowed. H318 Causes serious eye damage. H361 Suspected of damaging fertility or the unborn child. H410 Very toxic to aquatic life with long lasting effects.
Route of exposure	Skin, Inhalation, Ingestion, Eye	Skin, Inhalation, Ingestion, Eye
Frequency of use	Occasional	Occasional
Duration of exposure Minutes, hours	~20 mins	<5min
OELV[1] - ppm (TWA)	OELV - 8hrs (TWA)	N/A

[1] OELV = occupational exposure limit as set down in the most up to date Code of Practice for the Chemical Agents Regulations. If unavailable use TLV or equivalent