

**SERVICES & FACILITIES ANNUAL REPORT - FY April 2010 to March 2011**

|                         |                         |                               |                                       |                         |
|-------------------------|-------------------------|-------------------------------|---------------------------------------|-------------------------|
| <b>SERVICE</b><br>FENAC | <b>FUNDING</b><br>Block | <b>AGREEMENT</b><br>R8/H10/72 | <b>ESTABLISHED as S&amp;F</b><br>2009 | <b>TERM</b><br>End 2012 |
|-------------------------|-------------------------|-------------------------------|---------------------------------------|-------------------------|

**TYPE OF SERVICE PROVIDED:**

The Facility for Environmental Nanoscience Analysis and Characterisation (FENAC) provides a unique service meeting the needs of the 'environmental nanoscience' community. Originally envisaged as a service supporting the (eco)toxicological community investigating the biological impact of manufactured nanomaterials\*, FENAC also underpins the wider environmental community looking at manufactured nanoparticle chemistry and transport, along with nanoparticle investigations of other types of material sources including incidental (combustion, industry etc) and natural (microbial, weathering etc) nanomaterials. FENAC provides access and sample analysis for relevant samples, acting in a fully collaborative manner with FENAC users, helping users through the whole process from experimental design to data analysis. FENAC also provides training for doctoral and postdoctoral researchers during the sample and data analysis period and, more formally through 2 day summer schools, also leveraging NERC KE programmes and University of Birmingham support.

The FENAC Director is Professor Jamie Lead, with Dr Bjorn Stolpe the current Manager, supported by Dr Gillian Kingston (5%), with access through competition, with submissions due every 6 months. The facility offers a unique combination of experimental, analytical and metrological methodologies and the expertise to deploy such methods appropriately. Using a multi-method approach, FENAC incorporates a number of methods grouped as:

- microscopy (TEM, SEM, STEM, ESEM, AFM, CLSM),
- spectroscopy (EELS, X-EDS, XPS, FCS, ICP-MS)
- separation (FIFFF, SdFFF, CFUF, AUC, CE and disc ultracentrifugation) and
- other (DLS, XRD etc).

FENAC offers a unique, proven ability to characterise and interpret the physico-chemical properties of nanoparticles from all sources (below), including size, aggregation properties, surface behaviour, dissolution and morphology.

\*Nanomaterials are defined as having at least one dimension between 1 and 100 nm; NMs are of three types: manufactured (deliberately produced), incidental (accidentally produced) and natural (produced by natural sources).

**ANNUAL TARGETS AND PROGRESS TOWARDS THEM**

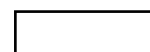
FENAC has made significant progress on all approved projects. Of 6 approved, 4 are complete, 1 is nearly complete and 1 is not yet started due to logistical issues with applicants. Given the high demand, significant method development has not been possible, requiring further staff or demand management, which has been discussed. Method development has been undertaken by other research projects led by the FENAC Director, ensuring FENAC remains internationally leading. FENAC has been widely marketed and availability disseminated at conferences, workshops and other venues. Demand is very healthy, particularly from the environmental and biological community interested in manufactured nanoparticles. A number of research papers have been published and substantial progress made in producing a review paper detailing the minimum characterisation required for nanotoxicology studies.

| SCORES AT LAST REVIEW (each out of 5) |            |                    | Date of Last Review:          |         |
|---------------------------------------|------------|--------------------|-------------------------------|---------|
| Need                                  | Uniqueness | Quality of Service | Quality of Science & Training | Average |
| 5                                     | 4.5        | 4.5                | 5                             | 4.75    |

| CAPACITY of HOST ENTITY FUNDED by S&F | Staff & Status   | Next Review (March) | Contract Ends (31 March) |
|---------------------------------------|--|---------------------|--------------------------|
| 20 %                                  | Professor Jamie Lead (3 hours per week); Dr Bjorn Stolpe (100% FT); Dr Gillian Kingston (5%FT) | 2014                | 2012                     |

| FINANCIAL DETAILS: CURRENT FY                                    |              |         |        |                   |           |                   |
|--|--------------|---------|--------|-------------------|-----------|-------------------|
| Total Resource Allocation<br>£k                                  | Unit Cost £k |         |        | Capital Expend £k | Income £k | Full Cash Cost £k |
|  | Unit 1       | Unit 2  | Unit 3 |                   |           |                   |
| 124.54   | 0.1          |         |        | 25.00             |           | 129.54            |
| FINANCIAL COMMITMENT (by year until end of current agreement) £k |              |         |        |                   |           |                   |
| 2010-11  | 124.54       | 2011-12 | 128.44 | 2012-13           | 2013-2014 | 2014-2015         |

| STEERING COMMITTEE | Independent Members | Meetings per annum | Other S&F Overseen |
|--------------------|---------------------|--------------------|--------------------|
| FENAC              | 6                   | 2                  | none               |



**APPLICATIONS: DISTRIBUTION OF GRADES (current FY — 2010/11)**

|                      | $\alpha 5$ | $\alpha 4$ | $\alpha 3$ | $\alpha 2$ | $\alpha 1$ | $\beta$ | R*/Pilot | Reject |
|----------------------|------------|------------|------------|------------|------------|---------|----------|--------|
| NERC Grant projects* |            |            |            |            |            |         |          | 1      |
| Other academic       |            | 2          |            |            |            |         |          |        |
| Students             |            | 1          | 2          |            |            |         | 1        | 3      |
| Pilot                |            |            |            |            |            |         |          |        |
| <b>TOTAL</b>         |            | 3          | 2          |            |            |         | 1        | 4      |

**APPLICATIONS: DISTRIBUTION OF GRADES (per annum average previous 3 financial years —2007/2008, 2008/2009 & 2009/2010)**

|                      | $\alpha 5$ | $\alpha 4$ | $\alpha 3$ | $\alpha 2$ | $\alpha 1$ | $\beta$ | R*/Pilot | Reject |
|----------------------|------------|------------|------------|------------|------------|---------|----------|--------|
| NERC Grant projects* |            |            |            |            |            |         |          |        |
| Other Academic       |            | 1          | 1          |            |            |         | 1        |        |
| Students             | 1          | 1          | 3          |            |            |         | 1        | 1      |
| Pilot                |            |            |            |            |            |         | 1        |        |
| <b>TOTAL</b>         | 1          | 2          | 4          |            |            |         | 3        | 1      |

**PROJECTS COMPLETED (current FY – 2010/11)**

|                      | $\alpha 5$ | $\alpha 4$ | $\alpha 3$ | $\alpha 2$ | $\alpha 1$ | $\beta$ | R*/Pilot |
|----------------------|------------|------------|------------|------------|------------|---------|----------|
| NERC Grant projects* |            |            |            |            |            |         |          |
| Other Academic       |            | 1          | 1          |            |            |         |          |
| Students             | 1          | 2          | 3          |            |            |         |          |
| Pilot                |            |            |            |            |            |         | 1        |

**Project Funding Type (current FY – 2010/11) (select one category for each project)**

| Grand Total | Infrastructure             |                      |       |             | PAYG  |                |                      |       |             |       |
|-------------|----------------------------|----------------------|-------|-------------|-------|----------------|----------------------|-------|-------------|-------|
|             | Supplement to NERC Grant * | PhD Students<br>NERC | Other | NERC<br>C/S | Other | NERC<br>Grant* | PhD Students<br>NERC | Other | NERC<br>C/S | Other |
| 10          |                            | 1                    | 6     | 1           | 2     |                |                      |       |             |       |

**Project Funding Type (per annum average previous 3 financial years - 2007/2008, 2008/2009 & 2009/2010)**

| Grand Total | Infrastructure             |                      |       |             | PAYG  |                |                     |       |             |       |
|-------------|----------------------------|----------------------|-------|-------------|-------|----------------|---------------------|-------|-------------|-------|
|             | Supplement to NERC Grant * | PhD Students<br>NERC | Other | NERC<br>C/S | Other | NERC<br>Grant* | PhD Student<br>NERC | Other | NERC<br>C/S | Other |
| 6           |                            | 1                    | 4     |             | 1     |                |                     |       |             |       |

**User type (current FY – 2010/11) (include each person named on application form)**

| Academic | NERC Centre/Survey | NERC Fellows | PhD Students | Commercial |
|----------|--------------------|--------------|--------------|------------|
| 19       | 3                  |              | 6            |            |

**User type (per annum average previous 3 financial years - 2007/2008, 2008/2009 & 2009/2010)**

| Academic | NERC Centre/Survey | NERC Fellows | PhD Students | Commercial |
|----------|--------------------|--------------|--------------|------------|
| 11       |                    |              | 7            |            |

**OUTPUT & PERFORMANCE MEASURES (current year)**

| Publications (by science area & type) (calendar year 2010) |     |    |    |     |     |       |             |          |                    |            |
|--|-----|----|----|-----|-----|-------|-------------|----------|--------------------|------------|
| SBA  | ES  | MS | AS | TFS | EO  | Polar | Grand Total | Refereed | Non-Ref/ Conf Proc | PhD Theses |
|  | 2   | 1  |    | 4   |     |       | 7           | 2        | 4                  |            |
| Distribution of Projects (by science areas) (FY 2010/11)   |     |    |    |     |     |       |             |          |                    |            |
| Grand Total  | SBA | ES | MS | AS  | TFS | EO    | Polar       |          |                    |            |
| 10   |     | 2  | 4  | 2   | 2   |       |             |          |                    |            |

**OUTPUT & PERFORMANCE MEASURES (per annum average previous 3 years)**

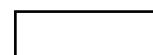
| Publications (by science area & type) (Calendar years 2007, 2008 & 2009)          |     |    |    |     |     |       |             |          |                    |            |
|---|-----|----|----|-----|-----|-------|-------------|----------|--------------------|------------|
| SBA   | ES  | MS | AS | TFS | EO  | Polar | Grand Total | Refereed | Non-Ref/ Conf Proc | PhD Theses |
|   | 2   |    | 2  | 2   |     |       | 6           |          | 1                  |            |
| Distribution of Projects (by science areas) (FY 2007/2008, 2008/2009 & 2009/2010) |     |    |    |     |     |       |             |          |                    |            |
| Grand Total   | SBA | ES | MS | AS  | TFS | EO    | Polar       |          |                    |            |
| 6   |     |    | 2  | 1   | 3   |       |             |          |                    |            |

**Distribution of Projects by NERC strategic priority (current FY 2010/11)**

| Grand Total | Climate System | Biodiversity | Earth System Science | Sustainable Use of Natural Resources | Natural Hazards | Environment, Pollution & Human Health | Technologies |
|-------------|----------------|--------------|----------------------|--------------------------------------|-----------------|---------------------------------------|--------------|
| 10          |                |              |                      |                                      |                 | 10                                    |              |

\*Combined Responsive Mode and Directed Programme grants

**NOTE:** All metrics should be presented as whole or part of whole number NOT as a %



## OVERVIEW & ACTIVITIES IN FINANCIAL YEAR (2010/11):

**General** Given the scale of the nanotechnology industry and the importance that NERC, other research councils, the EU and end users attach to the environmental and human health hazards and risks of nanomaterial, the start up of FENAC's operation has proved to be of great interest to the relevant NERC research community and to a range of governmental and industrial, both globally and nationally. Of more surprise, is that there is considerable interest in FENAC from researchers investigating incidental and natural nanoparticles. FENAC has raised the profile of UK nanoscience research globally, for instance contributing to at least one of the US-UK consortium awards based on the NERC ENI round, to an EU FP7 Infrastructure bid, and bids to the US NSF and Canadian NSERC. FENAC has also had discussions with organisations such as OECD and standards organisations (ISO, BSI). In part from FENAC's reputation, the Director has been invited by the BSI to write a Guidance Document related to the characterisation of NPs in complex media. Nationally, FENAC continues to work with applicants to NERC and other funding bodies and collaborated with a number of proposals submitted to NERC in this financial year. Details of the completed or current projects are given below. FENAC has also negotiated potential collaborations and links with the synchrotron facilities ISIS and DLS, with mutual benefits expected.

**Methods, Training and Staff Development.** The facilities available to FENAC at the programme start (April 2009) were substantial and are listed above in brief and the FENAC website in full. Further University of Birmingham funding has been leveraged to provide DLS, CE, BET, disc ultracentrifugation and FCS analysis (the latter with NERC support), with a value of ca £400 000. All instrumentation has now been installed and is routinely available.

As discussed above, major method development work has not been possible, although routine method development and training, along with method development by others in Lead's research group, has continued and informed FENAC practices, maintaining FENAC as an internationally leading centre. The issue with method development relates to the high demand for FENAC services. Either increased (technical) support, or demand management are likely to be required and these issues are under consideration and discussion. A number of planned method development areas are planned for the next financial year, including developments in FIFFF, TEM and reference materials.

Of the 6 projects approved, 5 have involved PhD students; 4 of these PhD students and an additional 2 postdoctoral researchers have been based at FENAC for training purposes for 1-3 weeks each, 9 weeks in total. In addition, we have run a second summer school attended by ca 15 doctoral and postdoctoral researchers (September 2010), with a third one planned for September 2011, at the Royal Society, London. The FENAC manager has been given specific instrument training and been involved in training others. User surveys are routinely distributed and indicate high satisfaction with the FENAC facility but will be used critically to ensure this level is maintained.

## SCIENCE HIGHLIGHTS

Collaboration with other Schools within the University of Birmingham ( $\alpha$  grade application) has resulted in productive research which is investigating the relative behaviour of standard and nano- hydroxyapatite from both industrial and microbiological sources. In particular, the wider project investigated the ability for hydroxyapatite to sorb trace metals for radionuclide decontamination in the nuclear industry. FENAC was requested to quantify the physico-chemical parameters of a range of hydroxyapatites to underpin and interpret the metal sorption data. SEM, TEM, XRD, BET and other data was collected which showed that factors such as crystallite size, specific surface area and organic carbon were key parameters in understanding why the nanohydroxyapatite from a microbiological source was the most effective metal sorbent. The work was performed in a collaborative manner, with the postdoctoral researcher on the application spending considerable time with FENAC for training. The project has already resulted in one paper being accepted to a leading journal, along with submission to the Goldschmidt conference (Prague, August 2011), which will lead to an extended abstract in *Geochimica et Cosmochimica Acta*. In addition, another paper is in preparation and a second application has been forwarded to FENAC for consideration in the May 2011 round.

## Publications

S. Handley-Sidhu, J.C. Renshaw, S. Moriyama, **B. Stolpe**, P. Yong, C. Mennan, S. Bagheriasl, A. Stamboulis, M. Paterson-Beedle, K. Sasaki, R.A.D. Patrick, **J.R. Lead**, L. E. Macaskie (2011). Removal of  $\text{Sr}^{2+}$  and  $\text{Co}^{2+}$  into Biogenic Hydroxyapatite: Implications for Biomineral Ion Exchange Synthesis. *Environmental Science and Technology*, 45, 6985-6990.

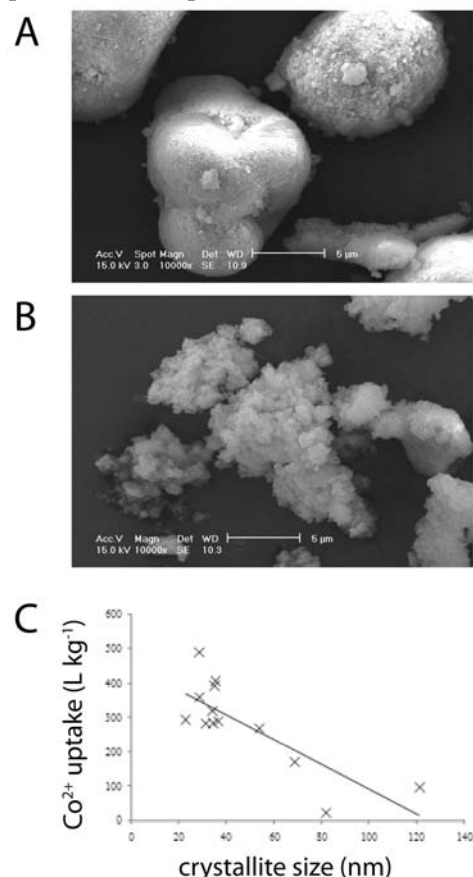


Fig. 1. SEM-images of hydroxyapatite (HAP), synthetic (A) and produced by bacteria (B).  $\text{Co}^{2+}$  - uptake was higher in bacterial HAP than in synthetic, and showed inverse correlation with crystallite size, determined by XRD (C).

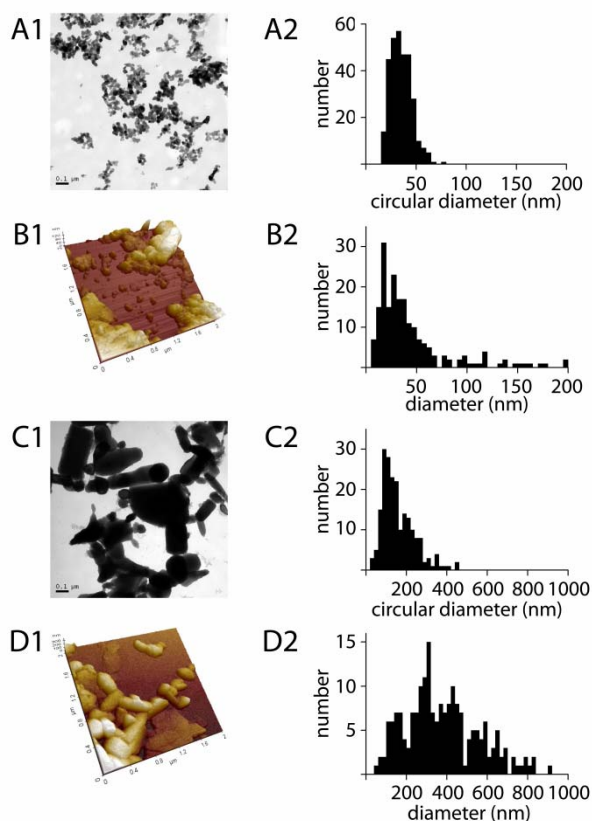


Fig. 2. ZnO nanoparticles (A, B) and bulk particles (C, D) studied by TEM (A, C) and AFM (B, D). TEM-circular diameter of discrete particles was  $35 \pm 10$  nm for nanoparticles (A2),  $160 \pm 80$  nm for bulk particles (C2). AFM-diameter (including aggregates) was  $50 \pm 60$  nm for nanoparticles (B2),  $380 \pm 190$  nm for bulk particles (D2).

### ions

J. Fabrega, R. Tantra, A. Aimer, B. Stolpe, J. R. Lead, C. R. Tyler, T. S Galloway (2011). Sequestration of zinc from zinc oxide nanoparticles and their effects on the sediment dweller and amphipod *Corophium volutator*. Environmental Science and technology (submitted).

A third project in collaboration with the British Geological survey has investigated the size and morphology of natural aquatic colloids from anoxic waters using AFM and TEM. Great care was taken in ensuring minimal perturbation of colloids and nanoparticles from their in-situ state was taken, with the AFM method in particular. Such a detailed AFM study on minimally perturbed anoxic materials has not been performed previously. Oxidation of the sample by controlled influx of air permitted an examination of the change in colloidal material caused by a change to oxic conditions. Two papers are in preparation, one based on methodological developments and one based on the hydrochemistry and colloidal chemistry of the particular catchments of interest. BGS plan to submit another application to FENAC in 2011.

### FUTURE DEVELOPMENTS/STRATEGIC FORWARD LOOK

FENAC will continue with the progress made to date in supporting the environmental and biological 'nano' community, in broadening its reach and access of the potential user community. Initial demand is high and FENAC will continue to meet these needs in particular in the NERC themes of Environment, Pollution and Human Health and of Technologies. The signs continue to be promising with applications to FENAC increasing and collaborations on NERC responsive mode and thematic programme applications. FENAC will continue to perform, improve and widen access to essential training via one-to-one laboratory training and summer schools, which also act as valuable outreach mechanisms.

Collaboration with the University of Exeter ( $\alpha 4$  application) resulted in a fruitful collaboration which has provided essential support to the OECD WPMN programme. The OECD has a list of 13 commercially relevant test materials which are being developed to underpin environmental and toxicological work on nanomaterial. By performing this research, FENAC has demonstrated important end-user impact through the project. The University of Exeter are contracted to perform ecotoxicological research on ceria and zinc oxide, research into which is being sponsored by the UK. FENAC has performed the physico-chemical characterisation on these samples, both as prepared and in relevant ecotoxicological media (size, surface properties, dissolution, and aggregation). Some analyses have been performed on zinc oxide after uptake to marine invertebrates (TEM and X-EDS to investigate sub-cellular compartmentalisation). Work has been undertaken in collaboration with the other Birmingham-based NERC facility (NBAF-B), which has quantified metabolomic signatures from zinc oxide NMs. The combined work at the two facilities and at Exeter represents a strong multi-disciplinary and intersectoral project, with high impact. One publication is in, several others are at an earlier stage of preparation (one extra purely from the FENAC data), while the University of Exeter has applied to FENAC twice more to continue the work zinc oxide and to extend to ceria.

### Publicat

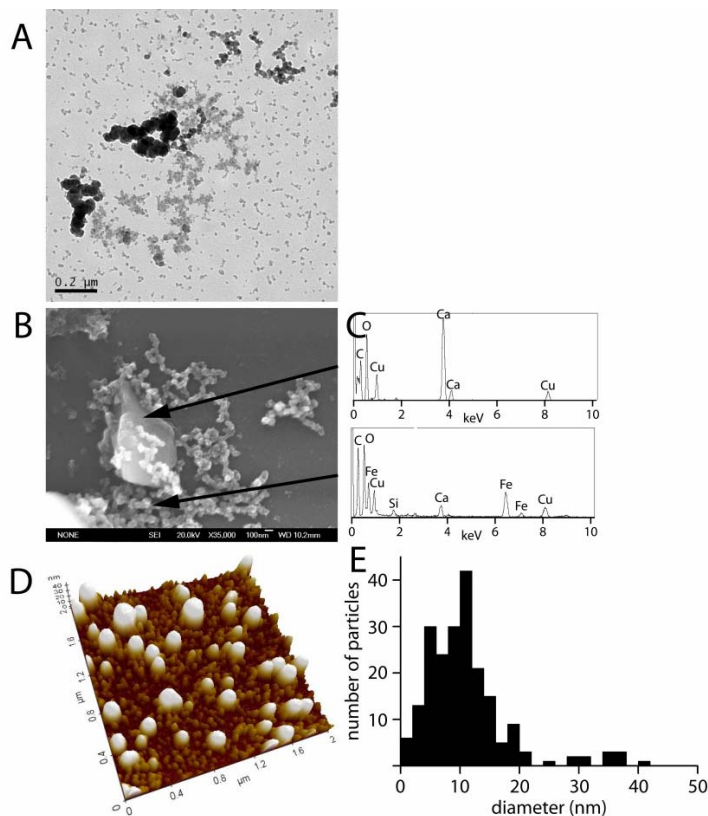


Fig. 3. Fe-rich nanoparticles, humic substances and Ca-rich particles in groundwater studied by TEM (A), SEM (B) and AFM (D). EDX-spectra show the presence of both Fe and Ca-rich particles (C). Average AFM-diameter of discrete particles was  $11 \pm 7.1$  nm (E).