RNME – REDE NACIONAL DE MICROSCOPIA ELECTRÓNICA

NATIONAL ELECTRON MICROSCOPY NETWORK

RNME – Pole of the University of Trás-os-Montes e Alto Douro

Unidade de Microscopia Eletrónica

ACTIVITY REPORT

2012

1. Global characterization of the Pole activity and progress.

In the period under consideration **25 different users (and 345 visitors)** required services of the UME at UTAD, divided by:

i) Training activities / Courses / Classes;
ii) Teachers / Researchers (PhD);
iii) Researchers (PhD under research grant contract);
iv) PhD Students;
v) MSc Students;
vi) BSc/License Students;
vii) Public and private companies;
viii) Maintenance.

2. Resources and operation conditions.

2.1. Material resources.

- Scanning electron microscope (SEM FEI Quanta 400, W filament), with environmental SEM / E-SEM and chemical analysis by EDS (EDAX), resolution of 4 nm.
- Transmission electron microscope (Leo 906 TEM 120 kV, W filament), resolution of 0.33 nm; conventional photographic acquisition system; in 2013 a CCD bottom mounted camera will be installed.
- Carbon deposition and gold sputtering (Polaron) for sample preparation;
- Ultramicrotome (RMC Power Tome XL) for sample preparation;
- Optical microscope and Magnifying glass (MOTIC), for sample preparation;
- X-ray diffraction (PAN'alytical X'Pert Pro, MPD) with X'Celerator detector, secondary monochromator; in 2011 it was installed an accessory for low angle acquisition;

2.2. Human resources supporting the experimental facilities operation.

According to UTAD regulations, the management of equipment and human resources (UME Technician) is made under the Foundation Rei D. Dinis / UTAD. The supervision of UME is made by two Senior Investigators of UTAD.

A technical commission of five investigators is responsible for formation, sample preparation, consulting, maintenance and guide-visits.

Scientific and technical resources

Name	Degree	Contract Category	Function in the RNME	RNME (h/week)
Ana Maria Nazaré Pereira	PhD	Full Professor	UME supervisor	2
Pedro Bandeira Tavares	PhD	Associate Professor	UME supervisor. SEM and TEM operator, formation, maintenance. Inorganic samples advisor.	12
Teresa Pinto	PhD	Auxiliary Professor	Biological samples preparation and advisor.	2
Paula Avelar Rodrigues	PhD	Auxiliary Professor	Ultramicrotome operator and formation Biological samples preparation and advisor.	2
Sandra Mariza Monteiro	PhD	Auxiliary Professor	Biological samples preparation and advisor.	2
Lisete Fernandes	MSc	Technician	UME Technician. Sample preparation SEM and TEM operator. Maintenance.	36

2.3. Rules and access conditions to the electron microscopy experimental facilities.

The requested services are subject to schedule, taking into account the constraints imposed by the operator (Technician of UME), maintenance or other obligations for the proper functioning of the equipment. The agenda should be carried out during the week prior to the completion of the work. The work from

Research Centers of UTAD has priority if the Technical Commission decides it. Biodegradable samples have also priority treatment.

Prices charged for services provided by UME are defined based on a table. This table and other relevant information are available at: http://home.utad.pt/~ume. The price for all RNME members is the same.

3. Training activities.

3.1 Training, information and demonstration activities addressed to researchers and to other users of the RNME.

The UME offers the possibility for teachers, technicians and researchers from UTAD to become operators of SEM. A course with 3 steps must be accomplished:

i) theoretical module (SEM and TEM);
ii) sample preparation module;
iii) operator module.

This last module represents a personalized instruction that involves specific training, with at least 12 hours of work in each equipment.

For the TEM (only after the SEM course), the formation is divided into two phases:

i) sample preparation (divided in biological and non-biological samples);
ii) operator module, with a minimum of 10 hours of individual training for each module.

In the final, an examination will take place that confers the certificate.

3.2 Teaching, training, information and demonstration activities in the frame of the University curricular activities

3.2.1 Training activities:

Training of equipment users/operators	Full Duration Days / Hours	Participants
SEM and TEM (sample preparation)	45h	Lisete Fernandes (Technician)
TEM (sample preparation)	12 h	Paula Avelar Rodrigues (Assistant Professor)
TEM (sample preparation)	6 h	Marco Lucas (Auxiliary Investigator)
TEM (sample preparation)	40h	Ana Paula (University of Coimbra technician)
SEM (sample preparation)	10h	Leonor Caldeira

3.2.2 Curricular units with training and teaching in electron microscopy unit:

Curricular units Course Discipline	Course Faculty / University	Number of Students	Number of hours
Materials	PhD in Quaternary, Materials and Cultures	8	6
Laboratory Techniques in Biology	PhD in Earth and Life Sciences	3	4
Instrumental Methods of Analysis	MSc degree in Laboratory Clinical Biology	20	2
Cell Biology	Degree in Applied Ecology	12	1
Electronic and Instrumentation	Degree in Biomedical Engineering	30	2
Ecotoxicology	Degree in Environmental Engineering	30	2
Advanced Topics in Materials	MSc in Mechanical Engineering	10	2

Geology	MSc Biology and Geology	4	2
Molecular Genetics and Biotechnology	Degree in Food Science	15	2
Cell and Molecular Biology	Degree in Medicinal Chemistry	15	1
	TOTAL	**147**	**24**

3.2.3 Study visits to UME (1 hour of visit per 20 students):

Activity	Promoter	Number of Students
Study visit Esc. EB 2,3 de Sta. Marta	R.P.I.	60
Study visit Instituto Politécnico de Bragança	R.P.I.	8
Study visit Esc. Sec. Morgado Mateus	UME	29
Study visit Esc. EB 2,3 de Sabrosa	R.P.I.	36
Study visit Esc. Sec. de Lamego	R.P.I.	60
Study visit Esc. Sec. Montalegre	R.P.I.	40
Study visit Esc. Sec. S. Pedro	R.P.I.	60
	TOTAL	**293**

R.P.I – Relações públicas e Institucionais da UTAD (UTAD Public Relations)
UME – Unidade de Microscopia Electrónica da UTAD (UTAD Electronic Microscopy Unit)

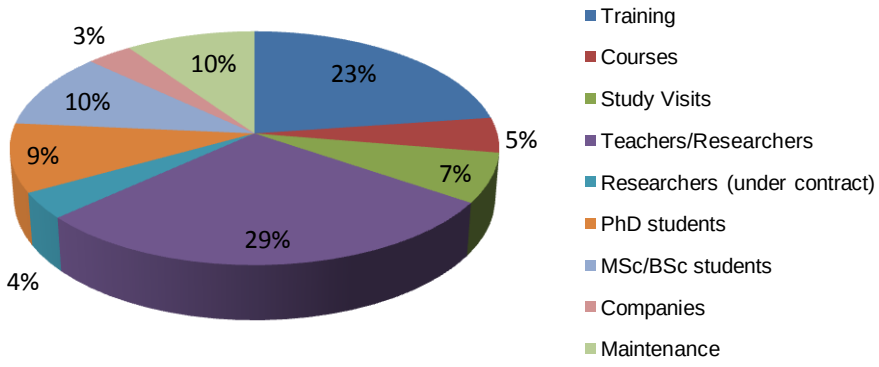
4. Electron Microscopy service activity and collaboration in research and development projects

4.1 Electron Microscopy service activity

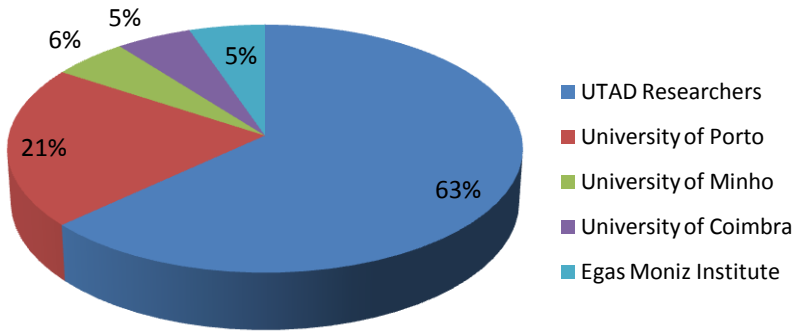
User Class Application	Number of Users	Number of Work Sessions	Number of Hours (h)	% of time
Training in EM	5	41	113	22.78
BSc, MSc and PhD Courses	147	10	24	4.84
Study visits	293	19	34	6.85

Teachers / Researchers (PhD)	15	65	143	28.83
Researchers (PhD) (under Research Grant contract)	3	10	18	3.63
PhD Students	8	12	47	9.48
MSc/BSc Students	4	11	52	10.48
Public and private companies (Industry and Services)	2	3	17	3.43
Maintenance	2	25	48	9.68
TOTAL	**479**	**196**	**496**	**100%**

Electron Microscopy Activity:



User's list by institution:



Teachers / Researchers (PhD)	SEM	Department Faculty	University Institution
Cristina Oliveira	✓	ECVA	UTAD
Pedro Tavares	✓	ECVA	UTAD
Tiago Pinto	✓	ECT	UTAD
Alexandra Esteves	✓	ECAV	UTAD
Fernando Nunes	✓	ECVA	UTAD
Marco Naia	✓	ECT	UTAD
Cristina Gonçalves	✓	ECVA	UTAD
Zilda Morais	✓	-	Escola Superior de Saúde Egas Moniz
Ramiro Fernandes	✓	ECT	UTAD
Paula Avelar	✓	ECAV	UTAD
José Almeida	✓	ECT	UTAD
Nuno Cristelo	✓	ECT	UTAD
Fernando Nunes	✓	ECVA	UTAD
André Pereira	✓	-	University of Porto
Margarida Marques	✓	ECVA	UTAD

Researchers (PhD) (under Research Grant contract)	SEM Equipment	Department Faculty	University Institution
Gopal Mishra	✓	ECVA	UTAD
Marco Lucas	✓	ECVA	UTAD
Fábio Figueiras	✓	-	University of Aveiro

PhD Students	SEM Equipment	Department Faculty	University Institution
Yonny Romaguera	✓	FCUP	University of Porto
Juliana Oliveira	✓	FCUP	University of Porto
Luísa Rodrigues	✓	Chemistry	University of Minho

Luís Silva	✓	ECT	UTAD/FEUP
João Pereira	✓	ECT	UTAD/FEUP
Cláudio Monteiro	✓	ECVA	UTAD
Lisa Martins	✓	ECVA	UTAD
Joana Rapazote	✓	ECT	UTAD

MSc Students	SEM Equipment	Department Faculty	University Institution
Ricardo Roma	✓	ECT	UTAD
Edgar Soares	✓	ECT	UTAD
Cláudia Fernandes	✓	ECT	UTAD

BSc Students	SEM Equipment	Department Faculty	University Institution
Leonor Ferreira	✓	ECVA	UTAD

ECVA – Escola de Ciências da Vida e do Ambiente da UTAD (Life and Environmental Science College of UTAD)
ECAV – Escola de Ciências Agrárias e Veterinárias da UTAD (Agronomic and Veterinary Science College of UTAD)
ECT - Escola de Ciências e Tecnologia da UTAD (Science and Technology College of UTAD)
FEUP - Engineering Faculty of University of Porto
FCUP- Sciences Faculty of University of Porto

Public and private companies (Industry and Services)
Serralves Foundation
ALCA

4.2 Publications generated or with reference to electron microscopy experiments made in this Pole of the RNME.

4.2.1. SCI Articles

- Célia Sousa, Diana Leitão, João Ventura, Pedro Tavares, João Araújo, (2012), *"A versatile synthesis method of dendrites-free segmented nanowires with a precise size control"*, Nanoscale Research Letters, **7 (1)** p.168 (7 pp) DOI:10.1186/1556-276X-7-168;

- Clara Pereira, André M. Pereira, Carlos Fernandes, Mariana Rocha, Ricardo Mendes, María Paz Fernández-García, Alexandra Guedes, Pedro B. Tavares, Jean-Marc Grenèche, João P. Araújo, Cristina Freire, (2012), *"Superparamagnetic MFe_2O4 (M = Fe, Co, Mn) nanoparticles: tuning the particle size and magnetic properties through a novel one-step coprecipitation route",* Chemistry of Materials, **24**, p.1496−1504;

- Gopal S. Mishra, A. Kumar, P.B. Tavares, (2012), *"Single site anchored novel Cu(II) catalysts for selective liquid-gas phase O_2 oxidation of n-alkanes"*, Journal of Molecular Catalysis. A, Chemical, **357,** p.125– 132;

- J.H. Belo, A.M. Pereira, J. Ventura, G.N.P. Oliveira, and J.P. Araújo, P.B. Tavares, L. Fernandes, P.A. Algarabel, C. Magen, L. Morellon and M.R. Ibarra, (2012), *"Phase Control studies in $Gd_5Si_2Ge_2$ Giant Magnetocaloric Compound"*, Journal of Alloys and Compounds, **529**, p.89-95, DOI: 10.1016/j.jallcom.2012.02.164;

- J. Oliveira, J. Agostinho Moreira, A. Almeida, V. H. Rodrigues, M.M.R. Costa, P. B. Tavares, P. Bouvier, M. Guennou, J. Kreisel, (2012), *"Structural and insulator-to-metal phase transition at 50 GPa in $GdMnO_3$"*, Phys. Rev. B, **85**, 052101 (5 pp);

- L. G. Vieira, J. L. Ribeiro, O. Santo, P B Tavares, (2012) "*Infrared anisotropy averaging in polycrystalline samples and resonant scattering: the example of $YMnO_3$*", Journal of Optics, **14**, 045707 (9 pp);

- M.Y. Teferi, V.S. Amaral, A.C. Lounrenco, S. Das, J.S. Amaral, D.V. Karpinsky, N. Soares, N.A. Sobolev, A.L. Kholkin, P.B. Tavares, (2012), "*Magnetoelectric coupling in multiferroic heterostructure of rf-sputtered Ni-Mn-Ga thin film on PMN-PT*", Journal of Magnetism and Magnetic Materials, **324** (11), p. 1882–1886;

- S.A.C. Carabineiro, P.B. Tavares, J.L. Figueiredo, (2012). *"Gold on Oxide-Doped Alumina Supports as Catalysts for CO Oxidation"*, Applied NanoScience **2**, (1) p. 35-46;

- S.A.C. Carabineiro, Nina Bogdanchikova, Pedro B. Tavares, José L. Figueiredo, (2012), "*Nanostructured iron oxide catalysts with gold for the oxidation of carbon monoxide*", RSC Advances. **2**, p. 2957-2965;

- Nuno Cristelo, Stephanie Glendinning, Lisete Fernandes, Amândio Teixeira Pinto, (2012), *"Effect of calcium content on soil stabilization with alkaline activation"*, Construction and Building Materials **29**, p.167-174;

- Nuno Cristelo, Stephanie Glendinning, Lisete Fernandes, Amândio Teixeira Pinto, (2012) *"Effects of Alkaline Activated Fly Ash and Portland Cement on Soft Soil Stabilisation"*, Acta Geotechnica, (*in press*).

- Pinto, J., Peixoto, A., Vieira, J., Fernandes, L., Morais, J., Cunha, V., e Varum, H., (2013), "*Render reinforced with textile threads*", Construction and Building Materials **40**, p. 26-32.

- Pinto, J., Cruz, D., Paiva, P., Pereira, S., Fernandes, L.,Tavares, P. e Varum, H., (2012) "*Characterization of corn cob as a possible raw building material*", Construction and Building Materials **34**, p. 28-33.

4.2.2. Other Articles with referee

- Benjamin Varela, Pedro Bandeira Tavares, Amândio Teixeira Pinto, João Castro-Gomes "Chemical Composition Correction of Aluminosilicate materials to Enhance their Conditions as Precursors for Alkaline Activation", Journal of Materials Science and Engineering with Advanced Technology, accepted for publication.

4.2.3. Books or Book chapters

- S.A.C. Carabineiro, A.M.T. Silva, G. Dražić, P.B. Tavares, J.L. Figueiredo, (2012), *"CO oxidation using gold supported on Ce-Mn-O composite materials"*, In: Carbon Monoxide: Sources, Uses and Hazards, Nova Publishers, Eds. D. DiLoreto, I. Corcoran, Nova Science Pub Inc., New York, p. 61-84 (ISBN-978-1-61942-055-7).

- T.T. Carvalho, J.R.A. Fernandes, N.A. Sobolev, V.S. Amaral, J. Agostinho Moreira, P.B. Tavares, (2012), *"Improvement of $BiFeO_3$*

multiferroic properties by chemical substitution allowing multifunction applications" (accepted for publication in Academy Publish).

- José Gomes-Laranjo, Lia-T. Dinis, Luís Martins, Ester Portela, Teresa Pinto, Marta Ciordia Ara, Isabel Feito Díaz, Juan Majada, Francisco Peixoto, S. Pereira Lorenzo, A.M. Ramos Cabrer, Changhe Zhang, Afonso Martins and Rita Costa, (2012), "*Characterization of Chustnut Behavior with Photosynthetic Trains",* Applied Photosynthesis, Mohammad Mahdi Najafpour (ed.), Published with Intech Open March, p. 47-80. (ISBN: 978-953-51-0061-4)

- BELINI, U.L., LOUSADA, J.L., PINTO, T., FERNANDES, L., TOMAZELLO-FILHO, M. (2012) - Anatomical characterization of sugarcane bagasse biomass for scanning electronic microscopy to wood panels. In IUFRO Division 5 – Forest Products Congress 2012, 8-13 julho, Estoril, Lisboa. 1:233.

- Lopes D., J. Lousada, L. Martins (CIFAP), J. Laranjo e T. Pinto (DEBA). 2012. Caracterização Dendrométrica e Fotossintética de Árvores Notáveis do Parque da Fundação de Serralves. UTAD, 70 pp.

4.2.4. Master thesis

- Ricardo André Dias Roma, "Melhoria do solo residual granítico de Guimarães com recurso à ativação alcalina de cinzas volantes", UTAD, 16/06/2012.

4.2.5 PhD thesis

- Yonny Romaguera Barcelay, "Propriedades estructurales, dinámicas y magnéticas de láminas delgadas de manganitas producidas por el método de sol-gel", Faculdade de Ciências da Universidade do Porto, 7/12/2012

- Carla Alexandra Orge Fonseca, "Tertiary treatment of effluents by catalytic ozonation", Doutoramento em Engenharia Química, Faculdade de Engenharia da Universidade do Porto, 21/12/2012.

4.2.6 Seminars

- Pinto, T., (2012). Preparação de amostras biológicas para análise por SEM. In: 16º Workshop SEMAT/UM – Caracterização de Avançada de Materiais (Técnicas de preparação de amostras para análise por

microscopia eletrónica – TEM, SEM, STEM. Universidade do Minho, Guimarães, 17 de outubro de 2012

- Monteiro, S.M., (2012). Preparação do Material Biológico para Observação em TEM. In: 16º Workshop SEMAT/UM – Caracterização Avançada de Materiais (Técnicas de preparação de amostras para análise por microscopia eletrónica – TEM, SEM, STEM. Universidade do Minho, Guimarães, 17 de outubro de 2012.

5. Analysis of activity progress with reference to planning, and strategies for improvement.

Due to budget limitations, in 2012, it was not affordable to sustain permanent maintenance contracts for the SEM (6500 Euros/year) or the TEM (8000 Euros/year). These contracts allow a rapid response for repairs (less than 72 hours) keeping the inactivity periods in a minimum. Without these contacts, the maintenance companies no longer assure a fast response, and sometimes take more than 2 months to answer a maintenance request.

1) For the period under consideration we have to refer a very important limitation of our SEM equipment due to the breakdown of the power source of the EDS detector (in March 2012). This damage, and the time that was needed to solve it, caused a considerable decreased in the number of hours of the microscope work as well as the number of users. The problem was solved in the beginning of June, with the acquisition of a new power source. However, another problem caused the failure of the EDS afterwards. The problem was solved by November 2012 with a replacement of a defected board in the EDS acquisition system.

2) The TEM has also a breakdown since November 2011. In this case, it is predicted to solve the problem by the end of February 2013, with the replacement of the touch screen and a board. This repair and a check up maintenance of the TEM will be performed by Zeiss (Barcelona Spain). At the same time the conventional photography acquisition system will be replaced by a digital CCD bottom mounted camera. This CCD camera was financed by FCT (55000 Euros) and an international contest was launched during December 2012 to supply this camera. The contest was won by Carl Zeiss and final report was already written. The installation is predicted for the end of February 2013.

In March 7, 2012, the work contract of our technician (Drª Lisete Fernandes) has expired. The Rector of Universidade de Trás-os-Montes e Alto Douro agrees in financing a one year technician grant (980 Euros/month). The call for a technician was published in Eracareers under the reference 1fdb5738-a313-44d4-a514-0b4809a0e66b. The selected candidate started the grant in May 1, 2012.

Vila Real, January 2013

Ana Maria Nazaré Pereira	**Pedro Bandeira Tavares**
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