Universidade da Beira Interior

Unidade de Investigação

MATERIAIS TÊXTEIS E PAPELEIROS

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ÍNDICE

PAPER GROUP Report

Task – 1. Papermaking potential

Considering the expected shortage of raw materials and aiming to diversify the sources for the pulp and paper industry, the potential of several raw materials, some of them invasive alien species, was studied. Some of them, such as Acacia melanoxylon, Acacia dealbata, and Ailanthus altissima, present good potential for pulping and papermaking, while others, like Cypressus, exhibited lower performance. Eucalyptus globulus, however, present the best performance in the hardwoods.

Task -2 Bioactive compounds

The screening of different shrub and wood species, as well as some industrial side streams, was carried out, in order to evaluate their potential as a source of biological active compounds. Crude extracts, fractions and pure compounds were screened for antioxidant, and antimicrobial activity. Essential oils of *Lavandula luisieri*, *Bulnesia sarmientoi* and *Schinus molle* were studied and their corresponding biochemical performance evaluated. The oils of *B. sarmientoi* and *L. luisieri* have shown natural insecticidal potential and the synergistic antibacterial activity of the essential oil of Aguaribay (Schinus molle L.) was established. Ailanthus altissima (Miller) Swingle, Acacia melanoxylon, Acacia dealbata and Olea europaea can be used as sources of bioactive compounds with antioxidant activity.

In carbohydrate chemistry, several syntheses of substrates for the preparation of new sugar derivatives and from these to synthesize new ferrocene glycoconjugates were conducted. The work was divided into 2 parts: synthesis of sugar derivatives and synthesis of ferrocene derivatives. In the first step, the hydroxyl groups of commercial monosaccharides were protected with suitable groups, namely benzyl acetate, and isopropylidene groups. The free hydroxyl group, which can be in position 3, 5 or 6 was oxidized to form an aldehyde or ketone group. From these compounds reactions to increase the carbon chain were made, using Wittig and Reformastky reactions. These substrates were linked to molecules containing ferrocene in its structure, thus obtaining the ferrocene glycoconjugates. The structures of these compounds were confirmed by spectroscopic methods and will be tested to determine their biological activity.

Task – 3. Release of sugars from vegetable biomass for bioethanol production

The scientific objective of this task is to contribute to a better understanding of the mechanisms determining sugar release from vegetal biomass, as well as the role of raw material physico-chemical characteristics. In a first approach, however, the potential of several wood and non-wood plant for bioethanol production was studied. The focus was put on the pretreatment stage required by lignocellulosic materials, and several possibilities were evaluated such as acid, alkaline, ammonium, and acid sulphite, including the effect of the operating variables. Diluted acid pretreatment of Cytisus striatus and Cistus ladanifer was optimized for bioethanol production. The effect of acid sulfite pretreatment on the sugar release of lignocellulosic materials through an enzymatic cocktail was also studied. HPLC was used to follow sugar, acid and inhibitory compounds produced in the pretreatments.

Task – 4. Cellulose nanofibrils

The group is extending their research activities to advanced cellulosic materials, including cellulosic based composites. Considering our laboratorial infrastructure and accumulated knowledge we are focusing on the rheological behavior of suspension containing isolated cellulose micro/nanofibrils or in combination with other natural polymers. On the other hand, we initiated the study related with the factors affecting the energy consumption in the production process and the micro/nanofibrillated cellulose quality. The work carried out aiming the liberation of sugars from lignocellulosic materials to bioethanol production has revealed the role that the enzymatic treatment may play in micro/nanofibrillated cellulose production. Only preliminary work was carried out in this field because there is no equipment available for the production of micro/nanofibrillated cellulose.

Task – 5. Fibrous materials functionalization

During this period, we worked in the surface modification of a commercial paper using cold plasma treatment in order to increase its hydrophilic character, thus minimizing the disintegration time and/or the energy consumption, needed to recycle cellulose fibers and obtain a homogeneous suspension. Cold-plasma-assisted treatment of additive-free hand sheet paper samples with acrylic monomers, styrene (**ST**), *para*-fluorostyrene (**FST**), *para*-fluoro-a-methylstyrene (**FMST**), *para*-chloro-a-methylstyrene (**ClMST**) and *para*-bromo-styrene (**BrST**) was studied and found that the grafting has occurred efficiently, as established by elemental analysis, contact angle measurement, and X-ray Photoelectron Spectroscopy (XPS).

Task – 6. Bleaching

A FCT project entitled “Study of compositional profile and seasonality of extractives in sapwood and heartwood from Eucalyptus globulus commercial trees - New approaches for reducing their influence in the pulping and bleaching processes” is being carried out. The projects aims to identify and evaluate the extractives in wood from Eucalyptus globulus, to study their variation with seasonality, tree age, and to develop a prototype to test pretreatments for extractives removal, in order to decrease their negative influence in pulp and paper production. The effect of tree age on extractives content was evaluated and enable us to conclude that extractives is not an obstacle to the use of older trees, but more chemicals in cooking and bleaching are required. The fiber morphological properties depend on tree age and position in the tree but all pulp samples exhibit very good papermaking potential.

Task – 7. Paper physics

The main objective of this FCT project is the development and the implementation of an optical method, based on retrodiffusion and transmission of the laser radiation, in order to measure and analyse the structural characteristics of the paper sheet, on a microscopic level. An experimental optical scanning system, for non-contact data acquisition, was developed and implemented to scan at once both sides of the paper sheets. The innovation of the proposed method is related with the fact that the measurements of the fiber orientation, in the surface and in the bulk, and the mass density are point correspondent for both paper sides. Papers with different fiber orientation and bulk densities were produced and are under evaluation.

Task – 8. Printing

In the printing field, a home-made advanced optical experimental setup allowed us to follow the ink drop dimension, their surface spreading and its final imbibition in paper. Several surface modified papers were also studied. This methodology contributes to understand the complex phenomena of the dynamic paper/ink interactions, contributing to the inkjet printing quality.

Task – 9. 3D Paper modelling

An innovative three dimensional paper model, where fibre flexibility, fibre dimensions and fibre collapsibility were introduced, was developed. The developed model gives realistic predictions for many paper properties, particularly the structural ones, for one type of fibres. The 3-D model of the paper are being extended in order to simulate the incorporation of two or more types of fiber, which significantly extend their potential. The potential to simulate fibrous nanomaterials is being explored. Computerized tomography was also used to analyze representative samples of different paper structures and the results are being evaluated.

Task – 10. Integrated Treatment of Cork Processing Wastewaters

The pollution load of cork boiling wastewater (CBW) samples was assessed through global parameters quantification, namely chemical oxygen demand, biological oxygen demand after 5 and 20-days incubation (BOD_5 and BOD_{20}), total phenols concentration (TP), etc.; and the concentration of several phenolic compounds, namely the gallic acid, protocatechuic acid, protocatechuic aldehyde, ellagic acid and tannic acid, were determined by HPLC. To assess the effluent molecular size distribution of the organic load, colour and TP fractions, the biodegradability (expressed in terms of the BOD_5/COD and BOD_{20}/COD ratios) and toxicity (MicroTox bioassay) we sequentially used four ultrafiltration membranes with molecular weight cut-off ranging from 10 to 100 kDa. Next, the molecular size fractions with >100 kDa, 50-100 kDa, 20-50 kDa, 10-20 kDa e <10 kDa were analysed before and after being submitted to ozonation with limited amounts of ozone ($O_{3,applied}$/COD ratios ranging from 0.15 to 0.60). We also analysed the molecular size fraction to have the concentration of the specific pollutants.

We also proceed with the adaptation of a CWL system with horizontal subsurface flow configuration to the CBW characteristics through increments of the organic load. The results were compared with a control bioreactor with the same characteristics, namely the use of LECA for the immobilization and development of the biomass but not planted with *Phragmites autralis*. Simultaneously, with the adaptation process we tested the viability of the integration of ozonation with the biological treatment and compared the results for the pre and post-treatment options, using an effluent with a COD concentration of 750 mg/L.

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TEXTILE GROUP Report

Objective - Development of wearable textiles prototypes, combining research in functional textile materials, incorporating sensorial and communication capabilities, aiming comparison of acquired signals to electromyography and ECG commercial devices.

N-type hydrogenated nanocrystalline silicon thin film piezoresistors, with gauge factor − 28, were deposited on rugged and flexible polyimide foils by Hot-wire chemical vapor deposition using a tantalum filament heated to 1750 °C. The piezoresistive response under cyclic quasi-static and dynamical (up to 100 Hz) load conditions is reported. Test structures, consisting of microresistors having lateral dimensions in the range from 50 to 100 μm and thickness of 120 nm were defined in an array by reactive ion etching. Metallic pads, forming ohmic contacts to the sensing elements, were defined by a lift-off process. A readout circuit for the array consisting in a mutiplexer on each row and column of the matrix is proposed. The digital data will be processed, interpreted and stored internally by an ultra low-power micro controller, also responsible for the communication of two-way wireless data, e.g. from inside to outside the human body.

In a research study, a vapor phase polymerization method, to synthetize coatings on fabrics for applications in smart textiles, is described. The method involves considerably less resources than traditional wet methods and used oxidant solutions of FeCl36H2O to synthetize poly(3, 4 – ethylenedioxythiophene) on fabrics of polyester. The method was optimized to maximize the electrical conductance of the coatings. The effect of the concentration of the oxidant solution in the electrical and mechanical characteristics of the samples was investigated and correlated to their morphology, analyzed by electron microscopy imaging. A strong decrease of the electrical resistance with the concentration of oxidant increase was obtained. A minimum sheet resistance value of 25 Ohm/sq was attained while the fabrics original mechanical tenacity was kept unchanged. The electrical resistance of the samples could be further decrease to 5 Ohm/sq by the application of multiple polymerization steps and was a function of the conjugated polymer content.

In the broad context of Wireless Body Sensor Networks for healthcare and pervasive applications, the design of wearable antennas offers the possibility of ubiquitous monitoring, communication and energy harvesting and storage. Specific requirements for wearable antennas are a planar structure and flexible construction materials. Several properties of the materials influence the behaviour of the antenna. For instance, the bandwidth and the efficiency of a planar microstrip antenna are mainly determined by the permittivity and the thickness of the substrate. The use of textiles in wearable antennas requires the characterization of their properties. Specific electrical conductive textiles are available on the market and have been successfully used. Ordinary textile fabrics have been used as substrates. However, little information can be found on the electromagnetic properties of regular textiles. Therefore this paper is mainly focused on the analysis of the dielectric properties of normal fabrics. In general, textiles present a very low dielectric constant that reduces the surface wave losses and increases the impedance bandwidth of the antenna. However, textile materials are constantly exchanging water molecules with the surroundings, which affects their electromagnetic properties. In addition, textile fabrics are porous, anisotropic and compressible materials whose thickness and density might change with low pressures. Therefore it is important to know how these characteristics influence the behaviour of the antenna in order to minimize unwanted effects. A research study presents a survey of the key points for the design and development of textile antennas, from the choice of the textile materials to the framing of the antenna. An analysis of the textile materials that have been used is also presented.

Fashion products, as for instance smart clothes or smart accessories, that integrate electronic components such as microcontrollers, tiny sensors and actuators, might be considered as assistive technology products. These smart fashion products may complement the function of a regular cane, helping the detection of obstacles in a very discrete and non stigmatizing way. The work of Leonardo Gontijo, consisting on the development of sensors to be hold in different parts of the body, has been reported in social media as a promising assistive technology solution. However, these type of products still remain unaffordable to the majority of the Brazilian visually impaired population. A research study presents the development of a cap that integrates a sensor of obstacles and an alert system. It is made of Brazilian sustainable materials and may be produced and commercialized at an affordable price. Some preliminary studies of its functionality are presented. By this way, the sensor cap might facilitate urban mobility, improve autonomy and help social inclusion of a large number of citizens. The work shows how design might promote social inclusion and might reduce stigmas, helping visual disabled people to live better.

Objective - Improvements on research on textile and clothing mass customisation.

A study results from an applied research project in collaboration with a textile firm located in Beira Interior. During this project the main objective has been the development of new patterns for wool fabric products for the company Fitecom. However, other objectives have been pursued, mainly the development of accessories with those fabrics, promoting differentiated applications beyond clothing products. Moreover, concerns about sustainability also support the work carried out until this stage. Concerning the raw materials, fabrics of natural fibers, 100% wool, were selected and for the design of the accessories, specifically bags, options of modularity were tested to extend the lifetime of the products, in a slow fashion perspective. The integration of technology, to increase the products value and consequently improve their lifetime, is also another solution proposed in this route to more sustainable products.

The development of fashion accessories is widely historically contextualized, as well as its relation to social changes and movements, in such a way that it is possible to identify and justify the emergence or development of a particular accessory according to the social and fashion context of a given period. The case of the bags is no exception and the relationship between clothing and bags have changed over time. Currently the majority of apparel brands develop parallel collections of bags that not only complement the clothing as well as increase brand value to the consumer. Starting from this association between clothing and accessories and the immense variety of materials that can be used in its creation, in this work the fabric as raw material for accessories, more specifically fabrics used in garment manufacture will be the main object of this study. This is an area barely documented in terms of fashion accessories design, however the constant need to create innovative and creative products, exceeding the conventional barrier is a fact, and the relationship between materials to apparel and accessories little exploited. It was used a literature review methodology within the area of historical evolution of bags and of the materials used and analysis of the usage percentage of textiles in accessories in some recognized brands in the Iberian market. It was also proposed the use of woollen fabrics in the production of this type of accessories. Finally was proposed the use of fabrics with yarns made of natural fibres and used mainly in apparel manufacture, whether in their natural state, either incorporating treatments and physical changes, can emerge as a way forward in the creation of differentiated products, increasing the offer available to the consumer, but also in terms of monetization of the same raw material by the company, but which assure performance requirements as other materials.

Modular design is considered by many as the key for a good design practice. Indeed, think products in terms of modularity as a tremendous potential, allowing the reliability and quality of the product, enabling maintenance, repair, simple assembly or disassembly, differential

consumption and also the reduction of production costs by companies when a family of products with the same modules is created. These characteristics can have a major impact in the product life cycle, improving extension of lifetime and reduction of waste and exploration of natural resources. In the research study is done a theoretical approach to modular design principles and practical methodologies and their implications on the life cycle of fashion design products, mainly in fashion accessories. Some prototypes developed under a research project on fashion accessories are presented as demonstrative of modular design application to the development of more sustainable fashion products.

Objective – Developments in product engineering design

A research work was based on an experimental concept of functional clothing for children with psychomotor development limitations. No matter the analyzed pathology, all these children need sensorial stimulation because of their psychomotor difficulties, especially at fine motor skills level. The main objective was to develop functional and comfortable clothing with sensorial stimulation elements (colours, textures, fragrances, sounds, etc.). It is intended, on the one hand, to increase the autonomy of the children in what concerns the act of dressing/undressing and, on the other hand, to stimulate their learning, coordination and self-esteem. A study about the specific needs of these children concerning clothing was worked out, which consisted in inquiring their parents and therapists. Based on the inquiries results, bibliographic revision in the area of therapeutic/interactive clothing and analysis of didactic and therapeutic material catalogues a clothing prototype (sweatshirt) was developed. The prototype was then tested by the children of the study sample and the test results were, once again, explained by the parents through the fulfilling of a prototype evaluation inquiry. This study supplied some important conclusions, more directed to the confirmation of the theme significance and to the definition of a methodology to be used in future research.

Industrially textured PLA multifilaments were heat set under different conditions at 110 ºC for 1 min and subjected to one cycle of deformation up to 20%. Thermal transitions of the original textured, heat set and cyclic strained filaments were measured using differential scanning calorimetry from 25 to 250 ºC. Cyclic deformation induces an endo-exo transition at peak temperatures between 55 - 60 ºC that is not observed in the original textured and heat set multifilaments. There is another endo-exo transition with peak temperatures between 67 and 72 ºC in all filaments, the intensity of which varies according to texturing conditions, heat setting and cycle strain. Texturing conditions, heat setting and cyclic strain play a role in thermal transitions, the strain at breaking and lastic properties of the filaments. The crystallinity and magnitude of the endo-exo thermal events detected by DSC are related to the strain at breaking and elasticity of polylactide textured multifilaments.

A research study reports an alternative approach for the evaluation of fabric pilling. On this approach, orthogonal projections of the three-dimensional (3D) fabric images are used directly to virtually evaluate the pilling formation of the fabrics. The 3D images of the fabrics are obtained through the reconstruction of the topography of the real textile fabrics. For that, a dual-scanning optical system (triangulation system) was used, which was developed and implemented by the research team. This evaluation approach is performed by comparing the appearance of the orthogonal projections of the 3D fabric images with the same photographic standard sets used in the conventional subjective method. For the validation of the obtained results with this methodology, a comparison was carried out relative to a reference subjective evaluation. This reference evaluation corresponds to a subjective evaluation of the fabrics originally performed by a panel of experts. A comparison was also performed between the results obtained with this methodology and those obtained by an objective evaluation. This objective evaluation

corresponds to the automatic attribution of the equivalent pilling grade to the fabrics, based on the calculation of the total volume of pilling formed in their surfaces. The final results obtained with the described methodology were showed to be globally in agreement with both the reference subjective and the objective evaluations. Subsequently, this method shows potential as an alternative approach for the virtual subjective evaluation of fabric pilling.

A research work reports on research being carried out in the area of functional knitted fabrics with a special structure incorporating hydrophobic fibres near to the skin and suction channels of hydrophilic fibres to suck moisture from the skin on the hydrophilic layer away from the skin; in this way, comfort is maximised in active wear as the fabric does not feel wet near to the skin.

Several factors affect the indoor air quality, among which ventilation, human occupancy, cleaning products, equipment and material; they might induce the presence of aerosols (or bioaerosols in the presence of biological components) nitrogen oxides, ozone, carbon monoxide and dioxide, volatile organic compounds, radon and microorganisms. Microbiological pollution involves hundreds of bacteria and fungi species that grow indoors under specific conditions of temperature and humidity. Exposure to microbial contaminants is clinically associated with allergies, asthma, immune responses and respiratory infections, such as Legionnaires Disease and Pontiac Feaver, which are due to contamination by *Legionella pneumophila*. Legionnaire's Disease has increased over the past decade, because of the use of central air conditioning. In places such as homes, kindergartens, nursing homes and hospitals, indoor air pollution affects population groups that are particularly vulnerable because of their health status or age, making indoor air pollution a public health issue of high importance. Therefore, the implementation of preventive measures, as the application of air filters, is fundamental. Currently, High Efficiency Particulate Air (HEPA) filters are the most used to capture microorganisms in ventilation, filtration and air conditioning systems; nevertheless, as they are not completely secure, new filters should be developed. The work aims to present how the efficiency of a textile nanostructure in a non-woven material based on synthetic textiles (high hydrophobic fibers) incorporating appropriate biocides to control *Legionella pneumophila*, is going to be measured. These bioactive structures, to be used in ventilation systems, as well as in respiratory protective equipment, will reduce the growth of microorganisms in the air through bactericidal or bacteriostatic action. The filter nanostructure should have good air permeability, since it has to guarantee minimum flows of fresh air for air exchange as well as acceptable indoor air quality.

In last decades, increased attention is paid to comfort properties of textiles and garments. The most important parameters characterizing the called physiological comfort of textile structures are the evaporative resistance and water vapour permeability. Contrary to common textiles, protective and functional garments and, also some technical textiles, are also used in wet state, which affect their comfort properties. A study was done and the PERMETEST commercial instrument is described, which provide reliable non-destructive measurement of evaporative resistance and water vapour permeability of fabrics in dry and wet state. By means of this instrument, evaporative resistance and water vapour permeability of cotton, polyester and cotton/polyester knitting fabrics, in the wet state, were experimentally determined and results were discussed. The effect of count yarn and composition of the above mentioned properties of these fabrics has been investigated as well. Some surprising results were achieved: with increasing fabrics moisture, the water vapour permeability also increases, especially with the presence of hydrophilic textile material.

Objective - Improvements on biotechnology and biomedical textiles.

In recent years, there has been an increase of infectious diseases caused by different microorganisms and the development of antibiotic resistance. In this way, the search for new and efficient antibacterial materials is imperative. The main polysaccharides currently used in the

biomedical and pharmaceutical domains are chitin and its derivative chitosan (CH) and alginates (ALG). With a simple technique of Layer by Layer (LbL) of applying polycation CH and polyanion ALG was used to prepare CH/ALG multilayers on cotton samples via the electrostatic assembly with success. The CH/ALG cotton samples (functionalized) were investigated for their antibacterial properties towards *Staphylococcus aureus* and *Klebsiella pneumonia* using the international standard method JIS L 1902:2002. The antibacterial activity of the functionalized samples was tested in terms of bacteriostatic and bactericidal activity, and results showed that the samples exhibited a bacteriostatic effect on the two bacteria tested, as expected. In addition, samples with five layers (CH/ALG/CH/ALG/CH) were more effective in inhibiting bacterial growth. This new coating for cellulosic fibers is a new strategy and may open new avenues for the development of antimicrobial polymers with potential application in health-care field.

A new biotechnological process that uses L-cysteine (L-Cys) which provides a permanent, nontoxic and effective antimicrobial effect over wool-based materials. This process is simple and carried out via widespread exhausting methods. Typically, wool fabrics are incubated with L-Cys for 50 min at 60°C in a pH 4.8 acetate buffer solution 25 m*M*, under mild agitation to give a good absorption rate. The minimal inhibitory concentration (MIC) of L-Cys was evaluated by the NCCLS M07-A6 standard method, and the results showed a good antibacterial activity against *S. aureus* and *K. pneumoniae* within the range of $6.0 \times 10^{-3} - 4.8 \times 10^{-2}$ g/mL [MIC 0.6% (w/v)] and $6.0 \times 10^{-3} - 4.8 \times 10^{-2}$ g/mL [MIC 0.6% (w/v)], respectively. In addition, the antimicrobial activity of the functionalized wool was assessed by the international standard JIS 1902-2002 showing a good inhibition of bacterial growth for an L-Cys concentration of 1% over the weight of fabric, both against *Staphylococcus aureus* and *Klebsiella pneumoniae*. Moreover, the biocidal mechanism was found to be related with the increase of sulfhydryl's groups onto wool fibers, which were quantified by the Ellman's reagent (5,5′-Dithio-bis(2-nitrobenzoic acid) method. The new process is easy to perform, non toxic, preserve wool quality and is a novel biomimetic approach that uses antimicrobial amino acids and may open new avenues for the design of biomedical textiles with a broad range of applications in healthcare.

Proteinaceous microspheres have a wide range of biomedical applications, including their use as drug delivery systems. On the other hand, bioactive and antimicrobial textiles are promising substrates for medical care, in particular, as wound-dressings. The research work relates the development of a new process for the functionalization of textiles through the simultaneous formation and linkage of protein-based microspheres onto textile fibers by sonochemical techniques. The microspheres developed by this process possess antimicrobial properties by themselves, but other may be incorporated by the encapsulation of various pharmaceutical formulations. This new type of microspheres and particularly their fixation onto textile materials encourage the development of textiles that can be used as delivery systems in a simple, fast, and non-toxic process. Here it is reported the production of microspheres with a combination of bovine serum albumin (BSA), L-Cysteine (L-Cys), and *n*-dodecane, using the ultrasound technology. The size distribution and morphology of the microspheres was determined as a function of several parameters such as irradiation time and BSA and L-Cys concentrations. The produced microspheres were analyzed using a laser light scattering size analyzer, an optical microscope and a scanning electron microscope. The new coating of BSA + L-Cys microspheres revealed a high stability and excellent antibacterial properties being a promising alternative to design textile-based bioactive delivery systems with potential application in the development of textile-based wound-dressings.

Synthetic fibers are used in various industrial applications such as packaging, protective coating, and biomedical and sealing materials. However, these polymers have few polar groups. As a result, they have a weak capacity to form hydrogen bonds with water (hydrophilic groups). Hence, low-pressure plasma treatments have been proposed to modify their surface properties (hydrophobicity and wettability) by introducing polar groups or by increasing the surface

roughness. In a research study, the focus was on a new approach to increase the hydrophilicity of poly(ethylene terephthalate) (PET) fabrics by a low-pressure plasma technique under three oxidizing atmospheres (oxygen [O_2] 100%, carbon dioxide [CO_2] 100%, and a mixture of O_2 (50%) and CO_2 (50%)). The plasma processes used in the study aimed to modify the superficial structure of polyester fibers, in particular PET, by forming new carboxyl, hydroxyl, and other polar groups on the surface. The increase in hydrophilicity was evaluated by the contact angles, the best results being 43.48° for the 100% CO_2 atmosphere and 44.56° for the combined CO_2 + O_2 atmosphere. It was also confirmed by the rising height, which was determined according to the International Standard DIN 53924. The results showed an important improvement in the hydrophilicity using both the combined CO_2 + O_2 atmosphere and the 100% CO_2 atmosphere. The surface energies of the fabrics were estimated using the contact angles. The presence of new carboxyl and hydroxyl groups was evaluated by staining methods using a cationic and a reactive dye, respectively. The results revealed an improvement in the water adsorption capacity due to the formation of carboxyl groups. Scanning electron microscope analysis of the surface morphology of the fibers showed an important cleaning action and oligomer elimination.

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Joana Vaz, UBI 2013 MSc in Biotechnology, "Estratégias de funcionalização bioativa de fibras celulósicas com péptidos antimicrobianos", Supervisor: Isabel Gouveia.

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Outros:

Local scientific coordination of project TEMPUS - 544390-TEMPUS-1-2013-1-GR-TEMPUS-JPHES - "***UNITE - University and Industry for the modernisation of fibres and textile manufacturing sector in Belarus***" – Março 2013

Team: TEI of Piraeus, School of technology applications, Department of textile, Universiteit Gent, VakgroepTextielkunde, Mogilev State University of Food Technologies. Faculty of Chemical Engineering Department, Belorussian State Economical University, Kaunas University of Technology, Vitebsk State Technological University, Faculty of artistic design and technology, CITEVE, IDEC S.A, Polytechnic University of Valencia, AITEX - Asociación de Investigación de la Industria Textil, and University of Beira Interior – (2013-2015)

ORIENTATION of RESEARCH STUDENTSHIPS, 2013-BI, MARTA PEDROSA (MICROBIOLOGY)
PROJECT: PTDC/EBB-BIO/113671/2009, SUPERVISOR: ISABEL GOUVEIA

Collaboration in 2 Prototypes featured on Modtíssimo (September 2013) of PT21

Collaboration in the development of Prototype 2-Smart Fashion-PT21 (under development)

Collaboration in the development of the Prototype Wearable Integrated Cardiovascular Surveillance System Project (under development)

Participação na Organização

Lectra Fashion, Wool Museum, 2013

Conference and workshop of fashion production-UBI, Covilhã

Festival IN November 2013

LISBON DESIGN SHOW October 2013

Modtíssimo, September 2013

Member of the Scientific Committee of the 2013 CIRCLE

Protótipos laboratoriais

Vest flag to Cyclist and Motorcyclist, integrating circuit with leds, batteries and solar panels, directed by Thayene da Silva Rezende, in the course of his Master thesis in fashion Design.

Patents
PPI 45857/12 - "Estrutura electrotêxtil multifuncional" - Quinta de S. Cosme Sociedade Agro-Industrial, unipessoal, LDA. - Nuno José Ramos Belino et al. – Submitted 2013 - PENDENT

PATENTES ACEITES

Isabel C. Gouveia e Daniela Sá, Patente PT 104540 (A) PROCESSO ANTIBACTERIANO PARA FUNCIONALIZAÇÃO BIOACTIVA DE MATERIAIS TÊXTEIS BIOMÉDICOS, GERIÁTRICOS E PEDIÁTRICOS, À BASE DE LÃ. (concedida em 2011)

Isabel C. Gouveia - PT 104823 "MÉTODO DE FUNCIONALIZAÇÃO BIOACTIVA DE MATERIAIS TÊXTEIS ATRAVÉS DA PRODUÇÃO E FIXAÇÃO SONOQUÍMICA DE MICROESFERAS PROTEICAS" (concedida em 2012).

FUNDAMENTAL SCIENCES GROUP Report

The work performed by the Fundamental Sciences Group is distributed by 4 different research lines (Cleaner Environment, New Stationary Supports for Affinity Chromatography, Rheology and Computational Fluid Dynamics and Green Chemistry in Medicinal Plants from Angola) and was developed according to 7 different research topics (RT). Below, the obtained results will be presented and discussed for each topic.

RT1: Advanced Oxidation Processes for the degradation of persistent pollutants

Regarding this research topic, and the objective FS1 (To perform the degradation of persistent organic pollutants from several type of effluents using advanced oxidation and membrane technologies), it was performed with success the anodic oxidation of azo dyes, aromatic amines with different substituting groups, pharmaceutical drugs, such as ibuprofen, tetracycline, and clofibric acid, among others, effluents from olive oil mils and leachates from sanitary landfills. These studies were performed with different electrode materials, namely, boron-doped diamond (BDD), PbO2 and SnO2-Sb, at a lab scale and with dyes and leachates from sanitary landfills they were also performed at a pilot scale. Texts were run in batch and in batch with recirculation modes.

In the work developed with leachates from sanitary landfills, leachates from two different sanitary landfills (Resistrela and Tratolixo) were submitted to electrocoagulation to reduce the organic load, prior to the anodic oxidation, and also to help in the precipitation of metallic ions, namely from heavy metals. In this combined treatment, it was also studied the influence of different experimental approaches on the increase in the biodegradability of the treated samples and on the removal of nitrogen forms, besides the removal of the organic load and metallic ions. These studies were performed with different types of effluents, and tests were also run with very good results at a pilot scale. Very high organic load removals can be achieved using electrocoagulation. However, ammonium removal is very low.

Leachates collected at three different sanitary landfills (Resistrela, in Fundão, Amarsul, in Palmela, and Tratolixo, in Abrunheira) were treated with anodic oxidation, performed with a BDD anode, using different approaches, namely, batch and batch with recirculation. The influence of the electrocoagulation pretreatment in the combined processes electrocoagulation+anodic oxidation was also assessed. The electrocoagulation pretreatment helps the increase in biodegradability during the anodic oxidation, meaning that a partial electrochemical treatment can be used to improve biodegradability of the leachates, allowing a biological treatment after the electrochemical combined treatment. Very good heavy metal removals were also achieved with the combined treatment.

Concerning the degradation of the pharmaceutical drugs and aromatic amines, it was also studied, by HPLC, the intermediates formed during the degradation. The use of combined processes, namely nanofiltration+ anodic oxidation, was also performed with very good results for the removal of phenolic compounds.

The research topic RT1 was also related with the objective FS2 (To prepare new stable oxide materials to be used as electrodes) and the work performed regarding this subject included the preparation of new stable oxide materials to be used as electrodes from tin and antimony oxides, over different substrates, namely titanium and titanium platinized, and from zinc oxide

and titanium oxide. These materials, after being prepared and characterized, were used in the degradation of azo dyes and pharmaceutical compounds.

RT2: Green energy and cleaner treated water

Under this research topic, different strategies were carried out in order to get a deeper understanding on anaerobic degradation of lignocelluloses compounds, proteins, polyphenols and dyes, among others, in order to produce a bio-fuel such as methane. Adsorption tests and anaerobic batch tests were carried out in a first phase.

These studies were mainly focused on the feasibility of biogas production and anaerobic remediation of pollutants associated with the agro-industrial sector, in particular phenolic aldehydes, in mesophyll batch systems. Depending on the type of substituting group, degradation may or may not occur. Addition of co-substrates seem to deeply affect the degradation rate.

RT3: Acid dissociation constants and solubility of dyes

Stoichiometric ionization constants of acetic acid were determined in the temperature range (10 to 40) °C, from potentiometric titrations in aqueous potassium chloride solutions with molalities up to 3 mol·kg^{-1}. The electrochemical cell was calibrated in terms of hydrogen ion concentration, and the calculations were performed by means of the SUPERQUAD computer program. The Pitzer theory, widely used in the evaluation of the excess free energy of nonideal electrolyte solutions, was applied to the calculation of the activity coefficient of each chemical species involved in the equilibria, and a good fit was observed at all temperatures. From these results, Pitzer interaction coefficients for potassium acetate were determined. This model was also used to calculate the chloride ion activity coefficient in acetate buffer systems with added potassium chloride, at ionic strengths higher than 0.1 mol·kg^{-1}, the limit of validity of the Bates–Guggenheim convention.

In order to assess the influence of the salt concentration on the acid-base equilibria, acid-base properties of azo dyes were also investigated and were determined both in water and in different ionic media (sodium chloride and sodium sulfate):

i) Ionization constants of methyl orange were determined by means of spectrophotometric measurements in water and in aqueous sodium chloride solutions with molalities up to 2 mol·kg^{-1} at temperatures between 278.15 K and 333.15 K. The temperature dependence of the thermodynamic acidity constant shows a slight curvature in accordance with published data. The influence of sodium chloride on the methyl orange deprotonation was assessed by the measurement of stoichiometric acidity constants in this ionic medium. The Pitzer theory, widely used in the evaluation of the excess free energy of non-ideal electrolyte solutions, was applied to the computation of the activity coefficients of the chemical species involved in the equilibria and a good fit of those equations to the experimental data was observed, at all temperatures under consideration.

ii) Acid orange 7 acidity constants were determined in water, in sodium chloride, and in sodium sulfate solutions, from spectrophotometric measurements, within the temperature range (278.15 to 333.15) K. The salt molalities were lower than (2 and 0.7) mol·kg−1 for sodium chloride and sodium sulfate solutions, respectively. Stoichiometric acidity constants were determined in those ionic media to assess the influence of those two salts on the acid−base equilibrium. Deviations from ideal behavior were estimated by the evaluation of the activity

coefficients of the ions that take part in the chemical equilibria by means of the Debye−Hückel theory. Expanded Debye−Hückel equations showed a good fit to the experimental data within the considered temperature range. The results also indicate the formation of dye aggregates at high ionic strength which is more evident as the temperature decreases.

RT4: Development of Organic Functional Dyes

The work developed can be described by the following items:

i) Synthesis of monolithics supports for affinity chromatography - For this purpose, poly(glycidylmethacrylate-co-ethylenedimethacrylate monoliths have been synthesized in different conditions (temperature, initiator, composition of porogenic solvent and concentration of crosslinking monomer). The preliminary results showed that the conditions of polymerization are crucial to the achievement of certain porosity.

ii) New N-carboxyalkylthiacarbocyanine dyes functionalized with amino derivatized groups as ligands in dye-affinity chromatography - Some derivatized dicarboxyalkylthiacarbocyanine precursors were synthesized envisioning the use of these new cyanines as ligands for Affinity Chromatography (AC). The introduction of three different groups in the 6- position of the cyanine benzothiazole nucleous require the previous reaction of 6-amino-2-methylbenzothiazole with decanoyl chloride, benzenesulfonyl chloride and phthalic anhydride to afforded 6-decanoyl-2-methylbenzothiazole, 6-benzenesulfonyl-2-methyl- benzothiazole and 6- phthalamide-2-methylbenzothiazole, respectively. Quaternary salts were obtained by the reaction of these compounds with 11-bromoundecanoic acid. Finally, the thiacarbocyanine dyes should be obtained by condensation of the resulting quaternary salts with triethyl orthoformate in the presence of pyridine.

iii) New supports for affinity chromatography by cyanine total synthesis in the chromatographic matrix – In previous studies, chromatographic supports were obtained by direct immobilization of the cyanine dye in the inert support (matrix) using a healing method. However, much remains unknown about the multi - variables responsible for ligand - protein interactions when cyanines are used in affinity chromatography. it seems to us fundamental to develop a method of immobilizing them in order to ascertain, step by step, the contribution of each party that, as a all, form a cyanine as a potential ligand for affinity chromatography. Thus, it were developed new chromatographic supports of derivatized cellulose with different types of cyanines, as well as its precursors at different steps of the construction of this dye. The different supports thus obtained will allow a subsequent individual chromatographic evaluation of the parts that make up this dye, namely hydrophobic spacer arm and heterocyclic ammonium salt.

iv) New N-alkyl-2-alkylsulfanylacetanilide atropisomers: synthesis and VT NMR study - NMR spectra of N-alkyl-acetanilides derivatives bearing a single alkylsulfanyl group in the ortho position indicate that they exist as two possible conformers, due to the restricted rotation about the Ar(C)-N bond. The corresponding rotation barriers were determined.

v) Critical temperature and free energy of activation evaluation of new 2-alkoxy-N-alkylacetanilide atropisomers - The synthesis of some atropisomeric ortho-substituted anilides 2 and their dynamic 1H NMR spectroscopic studies were performed. Dynamic NMR indicate that almost only two of the four possible rotamers are present in solution, with population ratios of 1:1. The measured free energy of activation to interconversion of the rotamers ranged from 18.85 to 19.16 kcal/mol.

vi) Re-examination of the base-catalysed hydrolysis of N-ethyl-2-methylbenzazol-3-ium iodides - The resulting unstable intermediates were isolated as the corresponding ether, thioether or selenoether derivatives, depending on the starting benzazole salt. Reduction of the o-(alkylchalcogen)acetanilides thus obtained afforded the corresponding o-(alkylchalcogen)anilines that are axially chiral molecules due to restricted rotation around the N-aryl bond. The resulting atropisomerism has been studied using dynamic VT NMR.

RT5: Rheology and Computational Fluid Dynamics

During manufacturing and processing many materials in the textile and paper industries exhibit non-Newtonian rheology, such as shear-thinning, normal-stress effects, viscoelasticity, thixotropy, etc. There is the need to be able to predict and understand the deformation and flow of such materials. So it is necessary the development of more robust and accurate numerical methods and techniques for the simulation of non-Newtonian flows and to acquire benchmark data to assess the numerical solutions and to enhance physical understanding of flow phenomena involving non-Newtonian materials. In order to fulfill these objectives numerical investigations of fundamental non-Newtonian flows were carried out with the following purposes:

i) Elastic instabilities in flows through expansions and cross-slots – non-Newtonian viscoelastic models induce flow asymmetries in situations where the corresponding Newtonian flow is symmetric. Cross-slot devices, in particular, are employed in microfluidic applications, for example to uncoil and count DNA molecules.

ii) Non-Newtonian flows in bifurcations – motivated by hemodynamical applications (genesis and location of atherosclerotic diseases). The size and intensity of vortices formed near bifurcating channels were quantified as a function of Reynolds and Deborah numbers.

iii)Algorithms for computational rheology – robustness of present methods was enhanced by implementing the log-conformation formulation for the stress equations and different algorithms were tested in time-dependent viscoelastic flows.

RT6: Preparation and study of new textile materials by nanomaterials incorporation

This study intended to prepare nanomaterials by wet or ceramics methods, followed by solid characterization of the synthesized materials. The prepared materials were then impregnated in textile substrates by corona, plasme or dip-coating methods. Environmental applications related to textile production processes and resulting wastewaters were also a target of this work. This work was partially developed in cooperation with the Textile Group and will be discussed in their report.

RT7: Medicinal Plants from Angola - biological activity

This research topic consisted on the harvesting drying and crude extracts preparation of medicinal plants from Angola. The extraction of the crude extracts was made against hexane, chloroform, ethyl acetate and water. The possible bioactivities of the extracts were also determined by bioassay guided fractioning with several cycles of fractioning and biological

activity evaluation of the obtained fractions. This study also included the isolation of pure biologically active natural compounds, MIC determinations, structural elucidation and evaluation of genotoxicity and cytotoxicity. Several plants were under investigation, namely, Croton gratissimus, Euphorbia conspicua, *Coriandrum sativum,* Alternaria solani, Eragrostis viscosa and Tinnea antiscorbutica Welv. The work developed resulted in several achievements, namely:

- Determination of the molluscicidal activity of compounds isolated from Euphorbia conspicua
- Determination of the antifungal activity of *Coriandrum sativum* essential oil, its mode of action against *Candida* species and potential synergism with amphotericin B
- Effect of plant extracts on growth and spore germination of Alternaria solani
- Isolation of three New Labdanes Isolated from Eragrostis viscosa
- Isolation of new neo-clerodanes from Tinnea antiscorbutica Welv

It was also studied the chemical composition of a new medicinal plant from Angola, the *Gymnosporia senegalensis*. The extraction was made with methanol that was fractionated hexane, chloroform, ethyl acetate and water. The extracts were fractionated by column chromatography with the appropriate solvents in order to obtain pure compounds of the lupano skeleton. The obtained compounds were characterized by IV spectroscopy, RMN 1D and 2D and mass sprectrometry.

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Organization of *International* conferences

I. Ferra and A. Lopes - - Member of the Scientific Committee and Charmain of sessions
18th Meeting of the Portuguese Electrochemical Society, 24-27 April 2013, Porto, Portugal
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Internationalization

P.J. Oliveira - Editorial Board of the ISRN Applied Mathematics, Hindawi Pub. Corporation. (Open access journal) (http://www.hindawi.com/isrn/appmath/editors/)

P.J. Oliveira - Member of the jury of the "Prize for the Best Luso-Spanish Doctoral Thesis in Rheology (from SPR- Sociedade Portuguesa de Reologia and GER- Grupo Espanhol de Reologia).

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