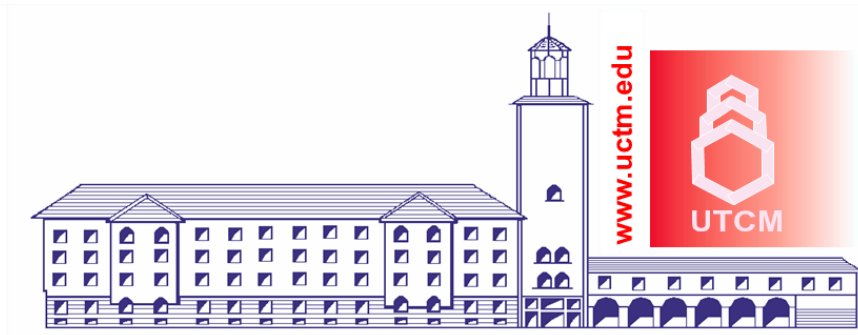


*25e anniversaire de la Filière francophone  
de Génie chimique et biochimique*



Université de technologie chimique  
et de métallurgie de Sofia

**ЮБИЛЕЙНА НАУЧНА КОНФЕРЕНЦИЯ С  
МЕЖДУНАРОДНО УЧАСТИЕ**

**„25 ГОДИНИ ИНЖЕНЕРНО ФРАНКОФОНСКО ОБУЧЕНИЕ  
В ХИМИКОТЕХНОЛОГИЧЕН И МЕТАЛУРГИЧЕН  
УНИВЕРСИТЕТ“**

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**DEVELOPMENT OF GC METHOD FOR CHOLESTEROL ANALYSIS IN FOOD**

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Levels of cholesterol in human body are extremely important because of its key role in many process in organism biochemistry. The total blood cholesterol, also named serum cholesterol level, has to be below 200mg/dL. If the level became of more than 240 mg/dL there is an increasing risk of cardiovascular diseases, ischemic heart attack, etc. Around 70% of cholesterol in organism is produced by liver concerning own needs of human body like vitamin and hormones production. Another around 30% comes in a human body by nutrition. It is very important to be possible to monitor the quantity of cholesterol coming from the external sources in the body. Herein, we report first steps of development of method for cholesterol determination using GC/FID. The calibration, linearity and choice of appropriate internal standard will be discuss. The obtained correlation coefficient was  $R^2=0,9991$ .

**Acknowledgments**

This work is supported by project 11688, 2017 of Scientific fund of UCTM.

### THREE-STAGE APPROACH FOR ISOLATION AND PURIFICATION OF *PSEUDOZYMA ANTARCTICA* LIPASE B

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Lipases are serine hydrolases defined as triacyl glycerol acyl hydrolases (EU 3.1.1.3). The unique characteristics and biotechnological potential of lipases have led to their use in food, detergents, pharmaceuticals, leather and textile industries, cosmetics and paper industry. Lipolytic enzymes can be isolated from plants, animals, bacteria, fungi and yeasts. In this work we report the synthesis, isolation and purification of the extracellular lipase from *Pseudozymaantarctica* NBIMCC 8340 using three-step process for isolation and purification. At every stage of the isolation lipase activity was analyzed by titrimetric and colorimetric method. In order to optimize conditions of synthesis and isolation two separate experiments of fermentation process, test and real one, were realized. Isolation and separation of lipase in our case were carried out by fractional precipitation with  $(\text{NH}_4)_2\text{SO}_4$ , followed by purification by ultrafiltration with micro concentration tubes Sartorius VIVASPIN 6. After useing of the two-step process of isolation and purification, was successfully isolated target *P. antarctica* lipase B. The resulting isolate was detected by SDS PAGE and analyzed for lipase activity. In order to obtain a lipase with a high degree of purity, as the third step was further conducted preparative isolation of the enzyme in native PAGE. After final step of isolation highly purified CalB with specific activity 92 U/mg was obtained.

#### Acknowledgments

This work is supported by project 11291, 2014of Scientific fund of UCTM.

**TAILORING GEMCITABINE BIOCONJUGATES TO SENSE AND KILL CANCER CELLS**

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Gemcitabine(dFdC) is an antimetabolite, a nucleoside analogue and is widely used as an anticancer agent against several solid tumors. Although its potency, it has two main therapeutic limitations: it lacks cancer cell selectivity and is rapidly transformed to an inactive uridine(dFdU) metabolite. To surmount these drawbacks we generated several novel gemcitabine bioconjugates with tumor homing peptides and their pharmacokinetic profile have been extensively explored. Furthermore, we constructed a theranostic molecular device that is able to sense and kill cancer cells integrating both photo regulated drug dosing properties and cancer microenvironment sensing characteristics.

**Topic :**Biochemistry and biotechnology

**VERIFICATION OF LABORATORY METHOD FOR ANALYSIS OF PHTHALATES IN FOOD AND BEVERAGES**

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1,2-Benzenedicarboxylic acid esters, which are commonly denoted as phthalates, form a Group of compounds that is mainly used as plasticisers for polymers such as polyvinylchloride (PVC). Because of their toxicity, phthalates are the subject of demand and determination in polymeric articles, including polymeric packaging of food and beverages, which are widely used in practice today. The migration of plastic components or additives from packaging to food can produce a risk for human health and that is the reason for developing and implementation of a method for determination of content of phthalates in different food matrices and beverages by using gas chromatography/ mass spectrometry (GC MS). A laboratory method for the determination of the following phthalates has been verified: within a range of 1 ppb to 1000 ppb for bis(2-ethylhexyl) phthalate, dibutyl phthalate, benzyl butyl phthalate, di-*n*-octylphthalat and a range of 20 ppb to 1000 ppb for diisononyl and diisodecyl phthalate. As an internal standard is used dicyclohexyl phthalate.

**Acknowledgment**

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**VERIFICATION OF METHOD FOR DETERMINATION OF BENZO[A]PYRENE IN FOOD MATRICES**

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Polyaromatic hydrocarbons (PAHs) are large group of organic substances. Some of them have proven carcinogenic effect on human's health. The development of industry and technologies leads to quick rising up of the level of different pollutants as well as PAHs. Sixteen of PAHs fall under consideration of European and International legislation for air, food and water. The need for quantifying of PAHs in different matrices increase very quickly.

In this study, we describe the verification of GC/MS method for determination of benzo[a]pyrene in two food matrices (flour and smoke low fat meat). We verified a set of performance characteristics (LOD, LOQ, precision (repeatability), recovery, linearity and specificity) together with evaluation of the uncertainty of measurement. Some developments in the method of sample preparation will be also discussed. The next step is to enlarge number of substances and type matrices we can determine by using this method.

#### Acknowledgments

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**TARGETED PEPTIDE THERAPEUTICS FOR TUMORS**

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Chemotherapy is one of the most common strategies for managing cancers. However most chemotherapeutics suffered from poor tumor targeting ability and high drug-originated side effects. As a result tumor site-specific drug delivery and penetration are two major challenges faced in chemotherapy. In this context, access to a drug delivery vehicle that could enable a simple and rapid multi-cargo installation, combining a cytotoxic warhead and a tumor adaptable homing element, could amplify the therapeutic potential of current drugs. Various peptide groups have been developed to overcome the penetrating and targeting problems such as CPPs (cell penetrating peptides) and TTPs (tumor targeting peptides). CPPs elevate membrane penetration, cell internalization and are widely used to enhance the intracellular delivery of various cargoes and nanoparticles while TTP peptides such as RGDs and NGRs are specific for tumor-related surface markers, such as membrane receptors, and can be used to deliver cytotoxic cargo (i.e. drugs or a cytotoxic peptide) specifically to tumor tissue or vasculature. Herein, we will present a single molecule that combines these two discrete features. We illustrate also that upon conjugation with a specific drug it enables the parent drug to fully enhance its original therapeutic profile, improving its cellular uptake and antiproliferative effect in specific cancer cells.

## STRUCTURAL CHARACTERIZATION OF A SYNTHESIZED PEPTIDE WITH POTENTIAL NEUROPROTECTIVE ACTIVITY AGAINST NEUROINFLAMMATION IN ALZHEIMER DISEASE

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Neuronal loss in Alzheimer disease leads to memory deficit as a result of a growing vulnerability to an asymptomatic state of progressive neuroinflammation [1]. Alzheimer's disease (AD) is characterized neuropathologically by formation of highly insoluble aggregates of amyloid-beta peptide. These plaques are co-localised with a broad variety of inflammation-related proteins such as proteic complement factors, acute-phase protein or pro-inflammatory cytokines [2]. Non-steroidal anti-inflammatory drugs (NSAIDs), such as aspirin, have been successfully used in reducing the risk of developing AD. However, NSAIDs drugs are not recommended for long-term use due to their side effects [3]. Experiments including co-incubation of neuropeptide type NAPVSIPQ and amyloid peptide fragments showed that NAP inhibited Aβ fibrillogenesis [4]. These neuropeptides are able to cross the blood–brain barrier and act as neurotransmitters in the intercellular communication [5].

Thus, we synthesized and characterized a new peptide which could have therapeutic properties in neurodegenerative diseases. The acetyl salicylic acid-NAP mutant, a potent drug in reducing neuroinflammation and plaque formation, was obtained using the well known F-moc solid phase peptide synthesis chemistry methods. The molecular mass was assessed by MALDI-ToF mass spectrometry and FT-IR spectroscopy.

**Keywords:** chemical synthesis, peptides, Alzheimer's disease, inflammation.

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## SOLID SYNTHESIS AND ANALYSIS BY MASS SPECTROMETRY OF FRAGMENTS DERIVED FROM AMYLOID- $\beta$ PEPTIDE AND THEIR COMPLEXES WITH VARIOUS METAL IONS

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Synthetic peptides have many practical applications and an important role in understanding and studying the properties of natural peptides [1]. For these reasons, there are always syntheses of new peptides and their structural analogs [2]. Recent research in this area focuses on the synthesis and characterization of reactions between the polypeptides and various compounds containing metal ions, representing a mutual problem in neurodegenerative diseases. The interaction between metal ions and peptides has been studied initially using spectroscopic or electrochemical methods such as circular spectroscopy (CD), NMR spectroscopy or cyclic voltammetry [3]. At present, mass spectrometry is a rapid method used more and more often in studies of proteins.

The main neuropathological feature of Alzheimer's disease is the extracellular deposits of  $\beta$ -amyloid peptides are associated with lipids, metal ions and other proteins. The development of neurodegenerative diseases focuses on abnormal accumulations of metals such as  $\text{Cu}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Al}^{3+}$  at the neuronal level, leading to the production of toxic protein aggregates [4].

Thus, two novel peptides modified with glycine and phenylalanine derived from the amyloid A $\beta$  (9-16) fragment were synthesized and characterized. Moreover, the peptides thus obtained have been subjected to interaction with various metal ions such as  $\text{Cu}^{2+}$ ,  $\text{Zn}^{2+}$  and  $\text{Ni}^{2+}$ . Both the structure of the manually synthesized peptides and their complexes with metal ions were confirmed by MALDI-ToF mass spectrometry.

**Keywords:** synthesis of peptides, A $\beta$ , metal ions, Alzheimer's disease, MALDI-ToF.

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**BIO-DEGRADATION WITH IMMOBILIZED CULTURE *PSEUDOMONAS*  
*DENITRIFICANS* IN CONTINUOUS MODE OF OPERATION**

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Biological denitrification has been used for years for waste water treatment. The potential of this method is great, reaching 100% water purification.

Experiments with immobilized cells of the strain *Pseudomonas denitrificans* were performed at various initial concentrations in continuous mode. It can be seen that a higher flow rate of the feed solution is possible at lower initial nitrate concentrations of the order up to 100 mg dm<sup>-3</sup>. The substrate concentration in the bioreactor outlet flow is related to the flow rate, precisely to the dilution rate (Dr). When increasing the concentration of nitrates in the feed solution, the dilution rate (Dr) at which breakthrough occurs becomes lower. In these cases the time for nitrate reduction is insufficient. The reason for this is the substrate inhibition of the denitrification process.

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**NATURAL PRODUCTS FOR DIABETES DISEASES TREATMENT**

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Diabetes is a chronic disease, which by inappropriate treatment methods, causes serious complications in body conditions and reduces the quality of people's life. Its spread is world-wide continuously increasing and it is expected that by 2025, 300 million people in the world will be affected by this disease. Diabetes disease is a metabolic disorder, characterized by a continuous increase in blood glucose above normal (hyperglycaemia) level to a gradual insulin secretion deficiency, based on insulin resistance. In Western countries, it represents 90-95% of all cases of diabetes. The incidence of diabetes disease increases usually after the age of 30.

High blood sugar can cause long-term complications, such as cardiovascular and kidney disorders, retinopathy, inadequate blood supply.

The development of diabetes can be prevented or delayed in people with impaired glucose tolerance by the use of lifestyle changes or the use of therapeutic agents. Some of these drugs are derived from plants or have microbial origin. Healing plants, as a source of natural products in various forms, are a new, better and more prevalent alternative for the prevention and treatment of patients suffering from diabetes, compared to available means and classical methods for disease treatment.

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**DESSIN, SYNTHÈSE ET ANALYSES DE NOUVEAUX TYROSINE-KINASE  
INHIBITEURS QUI AFFECTENT LE DÉVELOPEMENT DE LA LEUCÉMIE  
MYÉLOÏDE CHRONIQUE**

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**RESUME**

La leucémie myéloïde chronique (LMC) est une maladie myéloproliférative qui a caractérisé la prolifération autonome et incontrôlable d'ordres des cellulaires. Le traitement de la LMC supprime le défaut moléculaire responsable de la maladie. Ceci est obtenu par l'utilisation d'inhibiteurs de la tyrosine kinase. Le premier inhibiteur de la BCR-ABL-tyrosine kinase est l'Imatinib. Un problème majeur est que l'ABL-tyrosine kinase, il inhibe également la tyrosine kinase réceptive et un traitement prolongé avec Imatinib conduit à une anémie aplasique. En outre, de nombreux patients développent une résistance à ce sujet et appellent de nouveaux inhibiteurs. L'objectif de cette étude est la synthèse de nouvelles structures biologiquement actives sur la base de laquelle de nouveaux médicaments pour le traitement efficace de la LMC. Nous suggérons que la liaison à l'adénosine triphosphate de l'enzyme BCR-ABL-tyrosine kinase soit bloqué par des molécules de structure peptidique qui assurera la conformité spatiale avec la partie active de l'enzyme qui a ainsi une nature peptidique. Les oligopeptides à base de lysine ainsi que les peptides Phe-Lys-Lys-NH<sub>2</sub>, Phe-Phe-Lys-Lys-NH<sub>2</sub> par synthèse peptidique en phase solide, stratégie Fmoc sont synthétisés

**BIOBUTANOL PRODUCTION – RECENT ADVANCES AND FUTURE PROSPECTS**

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The increasing demand for alternative fuels employment due to natural resources depletion have forced scientists to explore different strategies in this direction. Among possible conventional fuel alternatives, may be the most promising is butanol, for its undoubtable advantages over the others known biofuels and the fact that it can completely replace the gasoline without any engine modifications. This review focuses on the biotechnological synthesis of butanol on the basis of wild and genetically treated organisms. It also focuses on the different renewable substrates that can be used for this purpose.

Concerning the economical point of view of butanol fermentation, of great importance is cheap renewable and non-food substrate utilization. For this purpose, various substrates are used: lignocellulosic materials, glycerol, syngas.

Biological butanol production naturally occurs in strains belonging to *Clostridium* genus and mainly – *Clostridium acetobutylicum*. This species is inconvenient to work with as it grows slowly, it is a facultative anaerobe and is very sensitive to butanol exposure. That is why butanol final concentration is too low ( $< 20$  g/L). These reasons make microbial butanol production economically unfavorable. To address these problems, numerous efforts have been made, as exploring different strains tolerance to butanol, cultivation media optimization, immobilization, co-culturing, genetic manipulation. Up to now the most promising strategy is molecular biology methods employment. The investigators manipulate the genes responsible for acids production and those, controlling solvents accumulation. Another possible approach is the insertion of butanol metabolic pathway in non-solvent producing host, which can survive its toxic influence.

## COMPARATIVE STUDY OF BIODEGRADATION OF 1,2-DIBROMOETHANE BY THREE DIFFERENT BACTERIAL SPECIES

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1,2-Dibromoethane (1,2-DBE) is synthetic organic chemical which is still produced in large amounts for use as a fumigant for stored grain; as a solvent for resins, gums, and waxes; and as an intermediate in the synthesis of dyes and pharmaceuticals. Its biodegradation passes through very toxic intermediates e.g. bromoethanol and bromoacetaldehyde. For a better understanding of the fate and persistence of 1,2-DBE in the environment and for the development of bioremediation techniques for the clean-up of polluted locations, it is important to study the physiology and ecology of bacteria that degrade this toxic compound. Batch mode experiments were carried out under aerobic conditions using sealed shaking flasks in a rotary shaker with 220 rpm at 30<sup>0</sup> C. In regard to biodegradation of 1,2-DBE by the strain *Xanthobacter autotrophicus* GJ 10 strong substrate inhibition is observed. Only slight part from substrate was utilized in 24 hours. In this time biomass reaches its peak and then its concentration drops down quickly. The dehalogenation capacity of the strain *Klebsiella oxytoca* NBIMCC 8391 is demonstrated too. Full degradation was observed at low and moderate initial substrate concentrations like 0,05, 0,08 and 0,1 g/l. The bromide ion release reaches its stoichiometric value when the concentration of microbial cells in the broth is the highest. Complete degradation at substrate concentrations as 0,1 and 0,2 g/l is observed by the strain *Bradyrhizobium japonicum* 273. At higher concentration as 0,3 and 0,5 g/l the substrate inhibition is strong and the biodegradation is getting more complicated. The cells exhibited long lag-phase and bromide were not released.

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**DETERMINATION OF PROHIBITED SUBSTANCES IN MODEL MATRICES****DÉTERMINATION DES SUBSTANCES INTERDITES DANS LES MATRICES  
MODÈLES**

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The authorities have identified an emerging trend where over-the-counter products, frequently represented as dietary supplements, contain hidden active ingredients that could be harmful. Consumers may unknowingly take products laced with varying quantities of approved prescription drug ingredients, controlled substances, and untested and unstudied pharmaceutically active ingredients. Hidden ingredients are increasingly becoming a problem in products promoted for sexual enhancement or weight loss products. The tests have revealed the presence of sibutramine, fenproporex, fluoxetine, etc. in weight loss products and the presence of sildenafil, tadalafil, vardenafil and their analogues in tainted sexual enhancement products.

The content of these substances is usually around the daily curative dose. Herein, first step is to identify if there are hidden active ingredients after that to determine their concentration per dose. The most important step is sample preparation in order to be sure that recovery rate of any possible hidden ingredient is appropriate so that to be detected. GC-MS has been regarded as a "gold standard" for substance identification. Sample preparation with derivatization step has been reported for spiked model herbal extract matrices.

**Acknowledgment**

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**ISOLATION AND PURIFICATION OF TYROSINASE FROM WILD MUSHROOMS  
(LEPISTA PERSONATA)**

**ISOLATION ET PURIFICATION DE LA TYROSINASE À PARTIR DE CHAMPIGNONS  
(LEPISTA PERSONATA)**

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The aim of this study was isolation and purification of tyrosinase from wildlife mushrooms *Lepista personata*. Enzyme was isolated and purified by precipitation with ammonium sulphate. The temperature, pH optimums and kinetic parameters were determined. Extract from *Lepista personata* were tested for antibacterial activity against to Gram negative and Gram positive microorganisms and results were discussed.

## OPTIMIZATION AND IMPORTANCE OF INERT CONDITIONS OF PRESSING OF PINK MUST FROM MAVRUD BY EXPERIMENTAL PRESS

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The oxygen in the wine making has an important role, that it is positive or negative. The exposition of the must or the wine with the oxygen decreases the quality and the varietal taste of the wine because of the oxidation, of the loss of the aromatic, the caramelization and the changes of the other characteristics. That is why the wine making of rose wines under inert gas (technology of wine making in absence of oxygen) takes more and more scale.

Years of experimentation with Mavrud grapes in "Centre du Rose" (Vidauban) and Lozev wine Ltd.(Svilengrad) show the impact of protection with azote and carbon dioxide in state pressing (method of inerting system). The absence of oxygen avoids the enzymatic and nonenzymatic oxidations, what leads to wines with more varietal characteristics, wealth in polyphenols more important and higher content of glutathion. Advantages of pressing under inert gas for the Mavrud grapes: Less use of sulfur; More aromatic, fruity and fresh Wines, (best notes in organoleptic analysis) provide from health must; Avoid the oxidation of the musts of rose wines (sensible state for rose must); Reduction of yellow color, the rose wine of Mavrud are less orange (method L,a,b); Increase of the content in phenols (acid hydroxycinnamique) and glutathion in the must. The acid hydroxycinnamique and the glutathion - two important antioxidants of the must - poliphénoloxydase they quickly oxidize in the presence of oxygen and of enzyme. The glutathion for the Mavrud grapes plays two important role, the prevention of the oxidation of musts and protection of quantity of resveratrol in the must ; the best conditions for the conservation of the varietal characteristics of Mavrud grapes.

Key words: dissolved oxygen, grape must, polyphenols, inert pressing

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**AN EXPERIMENTAL STUDY OF TEMPERATURE INFLUENCE FOR SWITCHING  
TRANSISTORS USED IN ELECTRONIC CONTROL UNITS FOR BLDC MOTOR  
CONTROL**

**UNE ÉTUDE EXPÉRIMENTALE DE L'INFLUENCE DE TEMPERATURE POUR LES  
TRANSISTORS DE COMMUTATION UTILISÉS DANS LES UNITÉS DE CONTROLE  
ÉLECTRONIQUE POUR LE CONTROLE DU MOTEUR BLDC**

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The operating temperature in automotive electronics industry depends by the following parameters: electronic control unit (ECU) location in the car, power dissipation of electronic components and thermal concept. That is the reason why an ever-present challenge for every electronic control unit hardware researchers and developers is selecting suitable components, such as MOSFET transistors, that not only perform effectively their intended or required function (behaving like switches) but also will survive without degradation or failure in time in high temperature operating conditions. For the safety point of view, there is a looming investigation: what will be the effects over the entire automotive system if the transistors used in ECU will get damaged due to temperature overstress?

The purpose of the paper is to demonstrate that temperature reached by automotive rated MOSFETs transistors used in powertrain transmission applications does not exceed maximum allowed junction temperature according to electronic components' manufacturer's specification. The transistors are part of an inverter used for a brushless DC motor (BLDC) control. The BLDC motor is an actuator used for automated transmission. For the measurements harsh automotive environment condition in terms of temperature is considered. Usually car's manufacturers require normal functioning even at 125°C ambient temperature. Two methods for motor control are evaluated: block commutation with rectangular currents form and sine commutation with sinusoidal currents form. In both cases, good results were expected.

## OPTIMISATION DE L'EXTRACTION D'ANTHOCYANINS DES DÉCHETS DE LA PRODUCTION DU VIN DE MAVRUD

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L'activité biologique des anthocyanines est principalement due à leur activité antioxydante et à leurs propriétés détoxifiantes qui viennent de leur capacité à bloquer l'action des radicaux oxydants libres. Les dérivés de la malvidine et de la cyanidine glucoside sont très présents dans les raisins noirs, y compris dans le Mavrud qui est un type de raisin traditionnel bulgare très ancien.

Dans cette étude, il s'agit d'optimiser l'extraction des anthocyanines provenant des pomaces et des pépins de raisins qui constituent les déchets de la production de vin de Mavrud situé dans la ville de Dimitrovgrad. Les échantillons ont été comparés avant et après l'extraction Soxhlet avec du n-hexane. L'influence de l'utilisation des ultrasons sur le rendement de l'extraction des anthocyanines a également été étudiée. Toutes les études cinétiques des extractions ont été réalisées avec des paramètres constants : 25°C, 100 min, solution d'éthanol à 50% dans l'eau et 0.1 g de solide par litre de solvant. La détermination de la concentration totale a été effectuée par la méthode du pH différentiel.

Les résultats obtenus montrent que l'application d'ultrasons augmente le rendement de 18% pour l'extraction des anthocyanines des pépins de raisins et de 15% celle des pomaces par rapport à l'extraction par agitation magnétique dans les mêmes conditions. Le dégraissage des échantillons extraits à l'aide des ultrasons permet également d'augmenter le rendement de 66.1% pour les pépins de raisins et de 101% pour les pomaces.

Il est essentiel de montrer un intérêt croissant pour le contenu anthocyanique des aliments et des nutraceutiques en raison des avantages pour la santé et de la récupération des déchets contenant les molécules bioactives.

Remerciements: Ce travail est soutenu financièrement par the Contract Grant DH 07/12

## STUDY OF THE RHEOLOGICAL PROPERTIES OF EMULSIONS BASED ON VEGETAL OILS

### ETUDE DES PROPRIÉTÉS RHÉOLOGIQUES DES EMULSIONS A LA BASE DES HUILES VÉGÉTALES

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Les dernières années sous la pression des consommateurs, de plus en plus de produits cosmétiques contiennent des extraits de plantes, le plus souvent des huiles végétales et des extraits huileux de plantes médicinales. Les émulsions sont la base de la formulation dans l'industrie cosmétique. La formulation des crèmes cosmétiques est déterminée par la fonction recherchée du produit (hydratant, mascaras, shampooing). Ces propriétés sont grandement influencées par les ingrédients mais également par les processus de fabrication. Les caractéristiques rhéologiques des cosmétiques sont importantes pour obtenir une qualité de haut niveau. Les aspects physico-chimiques et microbiologiques sont à prendre en compte pour la stabilité et la conservation du produit cosmétique. La sensation et la stabilité à long terme de la crème pour la peau, dans le cadre des applications cosmétiques sont des points importants pour le client.

Pour ce travail le comportement rhéologique des émulsions à base d'acide stéarique et contenant des huiles végétales (huile d'olives, huile de pépins de raisin, huile de graine de limnanthe) ont été étudiés. Le Triéthanolamine était utilisé comme émulsifiant et Sodium benzoate a été choisi comme conservateur.

Tous échantillons ont un comportement non-newtonien - liquides rhéofluidifiants, décrits par la loi d'Ostwald de Waele. Ces fluides ont leur viscosité qui diminue avec le cisaillement.

L'échantillon avec l'huile de graine de limnanthe a l'indice de consistance du fluide le plus élevé, et celle l'huile de pépins de raisin a l'indice d'écoulement le plus bas. Le changement dans les indices de consistance peut être expliqué avec la composition des huiles.

#### REMERCIEMENTS

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## DUAL-MODE DISTRIBUTED MODEL PREDICTIVE CONTROL OF A QUADRUPLE-TANK SYSTEM

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Model predictive control (MPC) has become the accepted methodology to solve complex control problems related to process industries. MPC involves the solution at each sampling instant of a finite horizon optimal control problem subject to the system dynamics, and state and input constraints. However, solving in a centralized way nonlinear MPC problems for medium- and large-scale systems may be impractical due to the topology of the plant and data communication and the large number of decision variables. Therefore, there is a strong motivation for development of methods for distributed solution of MPC problems.

Here, a *dual-mode* distributed MPC approach is proposed in order to reduce the on-line computational complexity of the distributed optimal control of *nonlinear* interconnected systems. It consists in using the *nonlinear* distributed MPC approach [1] when the state variables of the overall system are far from the origin and applying the *linear* distributed MPC method [2] in a neighborhood of the origin. The nonlinear distributed approach in [1] is based on first-principles (nonlinear) models of the interconnected systems dynamics. It includes a sequential linearization of these models and finding distributedly a suboptimal solution of the resulting quadratic programming problem. In order to apply the linear distributed MPC method in [2], it is necessary first to obtain a linearized model of the overall nonlinear system in a neighborhood of the origin. The benefit of the suggested *dual-mode* distributed MPC approach is the reduced complexity of the on-line computations in comparison to the entirely nonlinear approach when the current overall system state is in a neighborhood of the origin. The proposed method is illustrated with simulations on the model of a quadruple-tank system.

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## ANTIOXYDANTS CONTENU DANS LES POMACES DE RAISINS OBTENUS PAR DEUX MODES D'EXTRACTION DIFFÉRENTS

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La Bulgarie a un long passé viticole et est un des plus anciens pays producteur de vin au monde. Les déchets solides issus de la vinification pourraient représenter plus de 30% (w/w) des raisins utilisés. Les pomaces de raisin rouges, qui correspondent à la peau et aux pépins du raisin, représentent une source peu coûteuse de composés phénoliques précieux. Les composés phénoliques comprennent principalement les anthocyanines (la malvidine et la peonidine), les flavan-3-ol (la catéchine et les proanthocyanidines), les flavonols (la quercétine, la myricétine), les stilbènes et les acides phénoliques.

Dans cette étude la récupération des antioxydants provenant des pomaces et des pépins de raisins a été effectué par deux procédures d'extraction avec un agitateur mécanique et un magnétique. Pour les manipulations, les matériaux utilisés sont des pépins de raisins moulus (0.5 à 1.0 mm) et des pomaces bio provenant de la production viticole de Mavrud, récolté en 2016 dans la ville de Dimitrograd. Les extractions sont effectuées avec des paramètres constants : 25°C, 100 min, solution d'éthanol à 50% dans l'eau et 0.1 g de solide par litre de solvant. Toutes les analyses ont été effectuées par spectrophotométrie.

Les résultats obtenus montrent que la capacité antioxydante, la teneur en polyphénols, en flavonoïdes et en anthocyanines totaux, augmentent lorsque l'on utilise l'agitation mécanique pour les pépins de raisins et lorsque l'on utilise l'agitation magnétique pour les pomaces.

Les pomaces et les pépins issus du Mavrud sont des sources naturelles d'antioxydants et de composés phénoliques. Nous pouvons affirmer que le type d'extraction a un effet sur l'efficacité du processus et que l'optimisation est nécessaire afin que les bio-produits soient utilisés de façon économique.

Remerciements: Ce travail est soutenu financièrement par the Contract Grant DH 07/12.

## GREEN EXTRACTION OF HIGH ADDED VALUE SUBSTANCES FROM SPENT COFFEE GROUNDS:

### PRELIMINARY RESULTS

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Millions of coffee cups are consumed every day around the world, producing spent coffee grounds (SCG) in tons, which traditionally have been considered as wastes. Still, the SCG contain compounds of high added value as lipids, polyphenols, tocoferols and anthocyanins, and the recovery of these substances is very important in order to close the circle of food and agroindustrial residues re-usage.

The present work is devoted to comparison of the capabilities of supercritical fluid (SCE) and *n*-hexane extractions of SCG in terms of global yield, obtained under different conditions of temperature and pressure, and composition of the extracts obtained. The SCG from an espresso machine were obtained from a local coffee shop, then dried and stored in a refrigerator at 4° C until used. The SCE was performed in a laboratory apparatus equipped with a 50 cm<sup>3</sup> internal volume extractor, built from AISI 316 stainless steel tubing (32 cm long and an ID of 1.41 cm). The extraction was performed at  $T = (40 \text{ and } 60)^\circ\text{C}$  and  $p = (300 \text{ and } 400) \text{ bar}$ .

A slightly higher yield was achieved by SCE at  $T = 60$  and  $p = 300 \text{ bar}$ , while for the other conditions the yield was very close to that of the *n*-hexane extraction. Still, taking into consideration the experimental errors involved, it could be accepted that the performance of SCE with regard to the yield is similar to that of *n*-hexane extraction. With regard to antioxidant potential, the DPPH tests revealed that *n*-hexane extracts have higher activity than those of the SFE. Yet, the oils obtained by the two extractions have different colors, which can indicate that different compounds have been extracted. The latter is further supported by the preliminary results of NMR analysis of the extracts revealing differences in lipids composition.

Acknowledgement: The authors are thankful for the financial support of the National Science Fund, Ministry of Education and Science of Bulgaria under Contract Grant DH 07/12.

## GREEN SYNTHESIS OF SILVER NANOPARTICLES BY USING NATURAL PLANT EXTRACTS

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Nanotechnology is an up-to-date and interesting branch of science offering materials with structural features between those of atoms and bulk materials with at least one dimension in the nano range. Recently increase the number of research activities to explore properties of silver nano-sized particles. They may occur in different shapes as spheres, platelets, nanorods, dimeric nanorods, hexagonal discs, etc.

In this work, we present a simple, rapid and efficient green method for of silver nanoparticles synthesis. Silver nitrate (5 mM aqueous  $\text{AgNO}_3$  solution) was used as a precursor. Four different plant extracts: *Tilia* flowers; *Matricaria chamomilla* flowers; *Lavandula angustifolia* flowers, or *Calendula officinalis* flowers, were used as reducing agent and/or stabilizer. The adopted method can be easily implemented for any kind of scientific or industrial application due to its cost effective nature. The effect of the sunlight exposure on the obtained solutions was investigated. Silver nanoparticles were analyzed by transmission electron microscopy. The UV–Visible spectroscopy studies show the surface Plasmon resonance peak at between 440 and 470 nm characteristic of the spherical silver nanoparticles.

## SEMI-INDUSTRIAL PACKED COLUMN FOR INVESTIGATION OF LIQUID PHASE DISTRIBUTION IN HIGH PERFORMANCE PACKINGS

### COLONNE À GARNISSAGE SEMI-INDUSTRIELLE POUR L'ÉTUDE DE LA DISTRIBUTION DE LA PHASE LIQUIDE DANS DES GARNISSAGES HAUTE PERFORMANCE

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Packed columns are typical apparatuses for absorption, rectification and direct heat transfer processes with application in fuel production, gas and liquid purification, food industry and low grade heat recovery. The striving for sustainable industry and improving the efficiency nowadays have resulted in introduction into the market of new designs of high efficiency packings. The packed column's proper operation is strongly dependent on the regular distribution of the phases. The motivation of the present work is the lack of data on liquid distribution in a modern random packing Raschig Super-Ring, especially in industrial conditions, which is necessary for precise prediction of the processes in such packings.

A semi- industrial installation with 470 mm diameter packed column is reconstructed and equipped with the devices necessary for the liquid collecting method using tap water as a liquid phase. A liquid collecting device is placed under the packing layer consisting of 8 annular zones separated by concentric cylinders. A 5 mm wide zone adjacent to the column wall is used for measuring the wall flow. Two shower type liquid distributors are designed for different liquid loads covering together the range of initial superficial velocities from  $3 \times 10^{-3}$  to  $12 \times 10^{-3} \text{ m}^3 \text{ m}^{-2} \text{ s}^{-1}$ , which includes the typical regimes for industrial rectification and absorption. The liquid distributor is the device responsible for the initial uniformity of liquid distribution over the column cross-section, usually by feeding multiple streams of the same flow rate at equal distances. The presented liquid distributors have the same number of drip point 85 with orifice diameter 2,5 mm and 5,2 mm, situated at the tops of equilateral triangles with a 48 mm side. Two different densities of peripheral points are tested to achieve satisfactory initial uniformity.

**Keywords:** Packed bed column, liquid distribution, random packing, Raschig Super-Ring

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**EXPLORING THE POTENTIAL OF BLACK SEA MACROALGAE  
AS BIOENERGY (CARBOHYDRATE) RESOURCE  
EXPLORATION DU POTENTIEL DES MACROALGUES PROVENANTES  
DE LA MER NOIRE EN QUALITÉ D'UNE SOURCE BIOÉNERGÉTIQUE**

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Macroalgae derived from aquatic habitats of salty or fresh water are attracting worldwide attention for their potential as an economically sustainable source of carbohydrate bioenergy. Seaweeds (green, red or brown) contain various polysaccharides which represent great, but still unexploited resource for biofuel and chemical production. Macroalgae compare favorably with starch- and wood-based materials in respect to their limited lignin and cellulose contents, which makes the pretreatment and saccharification simple and easy. For conversion of the algal material into biofuels or chemicals, the hydrolysis pretreatment is an essential step which reduces the structural and compositional hurdles of the biomass in order to maximize the post-enzymatic degradation to monosugars.

Different pretreatment processes are known and could be applied; they include hydrothermal hydrolysis, steam explosion, acid or alkaline fiber expansion, soaking in aqueous ammonia or inorganic salts. Hydrothermal pretreatment, which does not involve expensive or hazardous chemicals, is the simplest, safe, economic, and environmentally benign method for the primary hydrolysis of the carbohydrates.

Here we present a study of green and red wild alga species harvested in the inter-tidal zones and on the rocks at the Bulgarian Black sea coast. The species are studied with respect to the carbohydrate yield that could be recovered therefrom and compared with cultivated red “Nori” algae. A technological scheme is developed, which allows a gradual release of the valuable algal components, starting from low thermal solid-liquid extraction to attain soluble sugars, polysaccharides, and proteins in the extract, to hydrothermal hydrolysis of the solid residue in order to obtain reducing sugars as a bioenergy resource.

## A NUMERICAL STUDY OF MASS TRANSFER IN A CROSS-FLOW FILTRATION CELL

## ÉTUDE NUMÉRIQUE DU TRANSFERT DE MATIÈRE DANS UNE CELLULE DE FILTRATION TANGENTIELLE

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The present study is part of a research in the area of integrated bioreactors with membrane separation, including the two possible configurations, inside submerged and side stream connected membrane modules.

The scope of the present work is a numerical study of the transfer processes in a side stream module. The aim is to reveal the characteristics of the mass transfer to the membrane surface in a cross-flow filtration cell, which is one of the factors directly affecting concentration polarization and the effective membrane operation. The other one is the shear stress distribution at the membrane surface, obtained in a previous study. By CFD simulation for the 3D geometry of the feed-side channel, the present work complements previous results on the flow pattern with data on mass transfer coefficient and its relation with the cross-flow velocity in line with experimental data from literature.

**Keywords:** cross-flow filtration, mass transfer, numerical simulation.

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## AN APPROACH FOR IDENTIFICATION OF FLEXIBILITY INDEX IN ATAD SYSTEMS

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In recent years Autothermal Thermophilic Aerobic Digestion (ATAD) of sludge has become a widely used technology for domestic wastewater treatment suitable for small towns and resorts. The ATAD system operates in batch mode, as aerobic microorganisms having exothermic metabolism are used for degradation of organic matter. A disadvantage of the ATAD system is deterioration of the operating temperatures in ATAD bioreactors due to thermal shock induced on the thermophilic microorganisms by charging each new portion of fresh sludge. Additionally, it is complicated by stochastic factors such as: temperature, amount and composition of the incoming fresh sludge, the hydraulic loading of the ATAD system etc. This problem can be overcome by redesign the ATAD system through recovery of the wasted heat for preheating the incoming raw sludge. Redesign problem is formulated as a multiscenario two-stage stochastic optimization which results in different solutions depending on the set of scenarios chosen. A flexibility index is used for decision-making which from the solutions obtained to be chosen. The flexibility index determines the size of stochastic space at which steady-state operational conditions of the system at pre-defined constant values of the design variables can be achieved. It determines the boundaries of the stochastic space in which the feasibility of the processes is ensured.

This study proposes an approach for flexibility index estimation. It consists in a definition of a central referent point regarding to which the boundaries of the stochastic space are scaled as the upper boundaries to take value 1, while the lower boundaries to have a value -1. The scaled space divides into subspaces as a hyper-rectangle is inscribed in each one of them. The sides of the rectangle stretch as long as a violation of the process feasibility constraints is not realized. The flexibility index is determined as a ratio between the sums of the volumes of inscribed hyper-rectangles and the volume of the whole stochastic space.

By the help of the defined in such a way flexibility indices, already obtained solutions for redesign of the energy-integrated ATAD system under uncertainties are estimated. Moreover, a decision is made regarding the most appropriate solutions in terms of process sustainability.

**Keywords:** Flexibility index, Stochastic optimization, Redesign, ATAD systems

**SYNERGISTIC AND ANTISYNERGISTIC EFFECTS OF  
ALIQUAT 336 AND TBP EXTRACTANTS USED IN ZINC RECOVERY  
EFFETS SYNERGIQUE ET ANTISYNERGIQUE DES SOLVANTS ALIQUAT 336 ET  
TBP  
APPLIQUES SUR L'EXTRACTION DE ZINC**

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With the development of industry the necessity for using metal increases still further. Hydrometallurgy plays an essential role in processing raw materials and industrial wastewater solutions. The liquid-liquid extraction, being basic part of it, aims to remove the components in aqueous feed solutions. In general, the organic phase contains an extractant, diluent and modifier. The main factors that influence the efficiency of the process include not only the organic phase composition, but also its concentrations. Sometimes when a mixture of two extractants is used, there is a deviation from the extraction additivity, which results from the chemical reaction in the organic phase. This deviation could be either positive, or negative. In the first case there is a synergistic effect, and in the second case there is an antisynergistic one.

The purpose of this study is to investigate the effect of the organic phase composition on the extraction of zinc from hydrochloric acid solutions with high chloride content through solvent extraction by varying the concentration of the commercial extractants – the solvating reagent TBP and anion-exchange quaternary ammonium salt Aliquat 336, and their mixture TBP/Aliquat 336 in the organic phase composition.

When comparing the extraction capacity of the individual extractants Aliquat 336 and TBP, it was found that pure Aliquat 336 exhibits a particularly strong extraction capacity towards zinc in terms of removing it through the physical extraction. Thus, the extracted amount of zinc by means of Aliquat 336 is significantly higher than the TBP used alone. In the mixed extractant TBP/Aliquat 336 there is a synergistic effect observed in the range below 10% of concentration in the two extractants, and an antisynergistic effect in the range above 10%.

## SYNTHÈSE DE GRAPHÈNE PAR PECVD : ETUDE EXPÉRIMENTALE ET MODÉLISATION

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### RESUME :

Ces dernières années ont connu une intense recherche sur les nanomatériaux où, le Graphène occupe une place privilégiée. Ceci est justifié par une conjonction de propriétés uniques modulables et conjuguables à l'infini. Propriétés mécaniques extrêmes, propriétés électriques, propriétés d'émission sous champs, grande stabilité chimique. Ces potentialités exceptionnelles justifient les nombreuses études expérimentales et théoriques consacrées au Graphène dans les laboratoires du monde entier. Ce travail de recherche est dédié à l'étude de la synthèse de graphène par le procédé de dépôt chimique en phase vapeur assisté par plasma micro-onde (PECVD) que nous avons abordé par une approche systémique du type "génie des procédés" alliant l'expérience et la modélisation. Il s'agit de dissocier un mélange gazeux composé d'hydrocarbure et d'hydrogène pour former des précurseurs carbonés. Ces précurseurs diffusent à leur tour à la surface d'un catalyseur métallique pour faire croître les nanotubes ou bien pour former des couches de graphène. Le travail de modélisation consiste à étudier la cinétique du plasma pour accéder à des paramètres difficilement mesurables expérimentalement. Nous avons développé une méthodologie expérimentale, théorique et numérique permettant d'analyser et de comprendre les mécanismes qui mènent à la croissance de Graphène. L'objectif final étant de maîtriser le procédé PECVD et proposer des matériaux à propriétés contrôlées.

Remerciements: La partie scientifique de ce travail est soutenu financièrement par le projet bilatéral « Modèles thermocinétiques pour la croissance de Graphène et de Nanotubes de carbone par dépôt chimique en phase vapeur assisté par plasma »

## MODÉLISATION NUMÉRIQUE DU COMPORTEMENT DE MILIEUX BIOLOGIQUES SOUS DES CHAMPS ÉLECTRIQUES PULSÉES

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L'exposition d'une cellule biologique à un champ électrique pulsé (CEP) caractérisé par des amplitudes de quelques kV/cm et de courtes durées (ms- $\mu$ s) conduit à un phénomène de perméabilisation membranaire. Une rupture locale dans la bicouche lipidique se produit en raison de la très faible conductivité électrique de celle-ci ( $\approx 1 \mu\text{S/cm}$ ) qui permet d'obtenir des hauts degrés de déstructuration des membranes cellulaires tout en étant rapides et à faible température. Les champs à l'origine de l'électroporation trouvent des applications innovantes dans le domaine médical mais également pour le traitement de la matière première végétale, associé par exemple à l'extraction par solvant des composés d'intérêt. Les mécanismes d'électroporation restent encore mal compris du fait de la complexité des phénomènes électriques, thermiques et biologiques qui sont impliqués. In addition, les solides d'origine végétale présentent une structure fragmentaire très hétérogène, de sorte que leurs caractéristiques électriques varient, et donc leur comportement lors du traitement électrique est très variable.

Afin de pouvoir prendre en compte les effets de la structure cellulaire, un modèle électrothermique a été développé, à l'échelle de la cellule végétale, constituée de plusieurs zones représentatives des milieux intérieur et extérieur d'une cellule, de sa membrane et la paroi qui les entourent. Ce modèle numérique, couplant l'équation de la chaleur à l'équation de Laplace, est résolu en 2D sur le logiciel de simulation par éléments finis COMSOL Multiphysics. La réponse a été comparée avec un modèle analytique 1D d'une cellule sphérique unique, composée d'une membrane et un cytoplasme homogène. La modélisation a confirmé que la membrane se charge, en particulier aux pôles de la cellule, dans la direction du champ, mais l'organisation du tissu a une grande influence sur l'amplification du champ électrique, le potentiel transmembranaire, l'énergie spécifique dissipée et l'élévation de la température. La comparaison a permis de mettre en évidence aussi l'effet de la paroi cellulaire sur le niveau d'électroporation, la paroi jouant également un rôle majeur dans le maintien de la structure cellulaire, sur laquelle le traitement CEP à effet thermique réduit a peu d'effets.

MOTS CLES :

modélisation FEM, champs électriques pulsés, perméabilisation, végétaux, extraction.

## EXTRACTION D'ANTIOXYDANTS A PARTIR DE PLANTES: L'ARTICHAUT ( CYNARA SCOLUYMUS L.)

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Les plantes représentent une source de principes actifs inépuisable et renouvelable, dont l'usage traditionnel et médical est connu depuis bien longtemps. Il existe donc un besoin de production de substances bioactives isolées, concentrées et purifiées, pour une utilisation dans un large champ d'application (cosmétiques, pharmaceutiques, additifs nutritionnels...), domaines dans lesquels la France est traditionnellement engagée. L'artichaut (*Cynara scolymus* L.) est une plante potagère, largement cultivé et consommé comme

légume dans les régions méditerranéennes. Riche en fibres, minéraux, vitamine B9, il possède de nombreux effets bénéfiques pour la santé. Cette plante est largement utilisée en médecine traditionnelle pour ses propriétés biologiques attribuées essentiellement aux polyphénols. Le bouton floral est la partie comestible de l'artichaut, mais ce sont les feuilles qui poussent le long de la tige, qui contiennent le plus de principes actifs. Afin d'évaluer la meilleure technique d'extraction de polyphénols totaux, de flavonoïdes et de tanins condensés d'artichaut, nous avons utilisé quelques techniques et méthodes d'extraction. Les procédés d'extraction sont basés sur la différence de solubilité des composés présents dans un mélange et dans un solvant. Nous nous intéressons ici à l'extraction à partir d'un système solide, la plante. Il existe plusieurs techniques d'extraction des produits à haute valeur ajoutée présents dans les plantes. Ces techniques peuvent être dites conventionnelles (utilisées depuis longtemps) et nouvelles (développées récemment).

## L'ABSORPTION DU DIOXYDE DE CARBONE PAR L'HYDROXYDE DE SODIUM

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### RESUME :

L'activité humaine augmente la composition chimique de l'atmosphère en dioxyde de carbone. Il y a beaucoup des méthodes pour débarrasser l'air de ce polluant toxique, l'une de ces méthodes est l'absorption du CO<sub>2</sub> par le NaOH. L'absorption est un transfert chimique qui existe lorsqu'on met en contact une phase gazeuse contenant une substance donnée et une phase liquide dans laquelle cette substance est soluble. La mise en contact de la phase gazeuse et la phase liquide se fait dans un contacteur gaz-liquide. L'un de ces contacteurs est la colonne à garnissage. Parmi les solvants chimiques, les solutions d'alcanoamines sont les plus couramment utilisés dans les procédés d'absorption des gaz acides. Le gaz réagit chimiquement avec l'absorbant liquide pour former des complexes plus ou moins faiblement liés.

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### MOTS CLES :

Absorption, lavage, gaz acides, alcanoamines.

## CONCENTRATION PAR ULTRAFILTRATION DE LA NISINE A PARTIR DE LA NISAPLINE 2,5%

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### RESUME :

L'ultrafiltration est un procédé de séparation en phase liquide qui s'effectue sous l'action d'un gradient de pression par des membranes permselectives en fonction des dimensions ou des poids moléculaires. L'objectif de cet expérience est d'étudier la faisabilité de l'ultrafiltration pour concentrer la nisine, un peptide antibactérien polycyclique, qui est utilisée dans l'industrie alimentaire pour prolonger la durée de vie des produits. Trois membranes possédants un seuil de coupure différent ont été testées. Des mesures des flux et des analyses sur le dosage des protéines ont été effectués afin de faire des conclusions sur la réussite de la méthode choisie..

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### MOTS CLES :

Ultrafiltration, concentration, nisine, membranes

## UTILISATION DE LA PERTRACTION (MEMBRANES LIQUIDES – LIQUIDES) POUR L'EXTRACTION DES ANTIBIOTIQUES

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### RESUME:

Les antibiotiques sont des molécules possédant la propriété de tuer ou de limiter la propagation des bactéries. Ils sont utilisés en médecine humaine et en médecine vétérinaire pour lutter contre des infections bactériennes et doivent être choisis en fonction de leur efficacité sur la bactérie à combattre, ce qui peut être testé grâce à un antibiogramme.

Séparation membranaire est un procédé permettant le transport sélectif d'une ou de plusieurs espèces chimiques entre deux phases séparées par une barrière sélective, solide ou liquide.

Ce travail concerne la récupération du phénol et de la tylosine d'une solution aqueuse diluée dans un pertracteur à films alternatifs tournants. Pour chacun des systèmes liquides triphasiques étudiés un modèle de transfert est proposé. Les coefficients partiels de transfert de masse et les constantes cinétiques des réactions chimiques mises en jeu sont estimés.

Les membranes liquides (solvants) utilisées sont le n-octanol et le n-décanol. L'influence du temps de séjour de la phase à raffiner sur l'efficacité de pertraction est mise en évidence. Le modèle proposé permet d'estimer le nombre d'étages théoriques nécessaire pour un taux d'extraction souhaité, et de déterminer les compositions des trois phases dans chaque étage.

Mots clés:

Pertraction, membranes liquides, extraction

**KNOWLEDGE-INTENSIVE BUSINESS SERVICES: FROM ENABLERS OF  
INNOVATION TO INNOVATION INDICATOR**

**LES SERVICES À FORTE INTENSITÉ DE CONNAISSANCES: D'UN FACILITATEUR  
DE L'INNOVATION A UN INDICATEUR D'INNOVATION**

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The research literature in innovation has constantly focused on the knowledge-intensive services (KIS) over the last two decades. Today, the smaller segment of knowledge-intensive business services (KIBS) has one of the highest developments in terms of creating jobs and providing inputs in the GDP and in the export in some of EU countries. Conceptually KIBS are defined as users, carriers and sources of innovation.

Innovation is key for the EU's growth and competitiveness. The need for innovation's rigorous measurement lead to the development and wide use of a range of approaches, methods and indicators. In October 2013, the European Commission proposed a new indicator to measure the innovation outputs and to benchmark national innovation policies and those of other European countries associated to the EU research Framework Program, and to monitor the EU's performance against its main trading partners. The competitiveness of the knowledge-intensive services is among the components of the new innovation indicator.

This article aims to contribute to the better understanding of the KIBS's role as a component of the new innovation indicator. The first part reveals the evolution of the KIBS's conceptual role of enablers of innovation for their client's to a formal indicator to measure the innovation outputs. The second section of the article critically discusses the presence of knowledge-intensive business services in the EU policies and their implementation in different countries.

**Keywords:** knowledge-intensive business services, innovation, innovation indicator, competitiveness, policy

## **ECO-INNOVATIONS IN BULGARIAN COMPANIES WITH PRO-ENVIRONMENTAL POLICY**

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The innovations play a significant role in accomplishing economic growth and bring additional potential for improvement of the business competitiveness. In the last years the innovations related to environment preservation are subject to ever growing interest by the companies and the society. In the same time the EU ecological policy through Environment Management systems (EMS) approach encourage and stimulate the companies to invest in eco-innovations. Therefore, the present paper analyses the relationship between EMS and the activity of the Bulgarian companies in development and introductions of new ecological technologies in the recent years. The conclusions are based on the results of a direct-individual-interview study conducted among Bulgarian companies from different sectors with implemented and functioning environmental management systems according to the international standard ISO 14001 and/ or EMAS. The obtained data revealed that the implementation of eco-innovations is in direct dependency of the economic sector and the market where the company operates as well as of the introduced regulatory requirements and the activities of the control bodies.

Key words: eco-innovations, Environmental management system, EMS, Bulgaria

**STUDENT-CENTERED LEARNING IN BILINGUAL ENGINEERING EDUCATION****ОБУЧЕНИЕ ОРИЕНТИРАНО КЪМ СТУДЕНТА ЗА ИНЖЕНЕРИ В  
БИЛИНГВАЛЕН КУРС**

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The educational approach for student- centered learning (SCL) is applied to develop engineering and communicative skills in biotechnological students. A new educational model has been developed, it is based on: students' needs reporting, use of authentic professional tasks and materials in bilingual form; collaboration and team work, learning activities that reflect the professional duties of specialists in their workplace; specific achievement assessment (academic knowledge, skills and communicative competencies).

Bilingual environment has been developed through integration of the English language course and the respective technological content. The idea is to create tuition that passes from learning 'English for specific purposes' to 'communication for specific purposes'. With that end in view specific educational materials have been created and a flexible activity system for lecturers and students has been applied.

In order to assess the effects of the innovation, the learners' achievements and the levels of satisfaction have been measured in different components. Here we discuss the potential of bilingual learning environment to provide SCL according to the data from a self-esteem survey for students.

## **“GREEN” COMMUNICATIONS AND CORPORATIONS**

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A research focus is forms of so-called "green" communications with which corporate sector transmits messages related to sustainable development concept to its stakeholders.

The study is guided by the most widely accepted sustainability definition: "A development that meets the needs of the present without compromising the ability of future generations to meet their needs" (Report of the World Commission on Environment and Development: "Our Common Future" 1987, known as the Bruntland Report). However, other sustainable development perspectives are also presented, showing a broad conceptual framework.

The article also discusses basic interpretations of “green” corporate communications, such as: the utilitarian interpretation of these communications as a "marketing tool"; company's products diversification; responding to existing social pressure; company's values expression, etc.

As stakeholders expect local and multinational companies to implement "green" communications, the corporate sector operates in a new environment of altered communication. Different corporate communications strategies and tools for sustainable development are examined in the presente papier. Due of the diversity of approaches, the article also highlights some key researchers' attempts to categorize the corporate communication practices types in sustainability area.

Key words: “green” communications, “green” corporate communications strategies, “green” corporate communication practices

**„GREEN“ BUSINESS MANAGEMENT AS SME BUSINESS OPPORTUNITIES**

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The report analyses the opportunities for „green“ management of organisations. The paper is focused on the activities of small and medium-sized companies in Bulgaria, as well as their views and problems for the development of "green" business management. The survey analyzes the views of the participants in the focus group on the definition of green business; the specifics of "green" activities and their financing; strategies for internal and external communication of "green" governance; content offerings for "green" business management training focused on small and medium businesses.

Key words: “green” management, business opportunities, small and medium enterprise, SME, Bulgaria

## ERGONOMIE ET QUALITÉ

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### RESUME :

L'ergonomie et la qualité ont leur propre discipline. Il existe de nombreuses similitudes entre les deux. Les relations entre l'ergonomie et la qualité ces dernières années ont été prises en compte. Par conséquent, il y a peu d'informations dans ce domaine. Il n'y a pas assez d'informations sur la façon dont divers aspects de l'ergonomie peuvent avoir une influence sur la qualité. Clarifier la relation entre les deux questions n'est pas seulement utile pour les gestionnaires qui se concentrent davantage sur la qualité, mais aussi pour les ergonomes.

**MOTS CLES :** ergonomie, qualité, amélioration continue

## PRELIMINARY STUDY ON AN ION CHROMATOGRAPHIC METHOD FOR ANION DETERMINATION

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Ion chromatography is a well-established method for determination of anions in water samples. However, to ensure precise and reliable results the method should be optimized and validated in each laboratory.

This report presents the results from a preliminary study of an ion-chromatographic method for determination of fluoride, bromate, chloride, nitrite, bromide, nitrate, phosphate and sulphate in natural water. The method characteristic working range, linearity, limit of detection and limit of quantification were studied according to the international regulations for method validation.

An ion chromatograph equipped with autosampler, the samples filtration system with a 0.2  $\mu\text{m}$  membrane, injection valve, high-pressure pump, suppressor module and conductivity detector was used. The separation was performed on Metrosep A Supp 7-250 (Metrohm) anion separation column, with 3.6 mmol/l  $\text{Na}_2\text{CO}_3$  as eluent in isocratic mode at flow rate of 0.7 ml/min, temperature 45 °C and injected volume of 20  $\mu\text{l}$ .

The obtained experimental results showed that at the selected conditions the ion chromatographic method allowed determination of low concentration of targeted anions in waters. The limit of detection (LOD) were 0.7, 7, 10, 6, 2, 0.8 and 6  $\mu\text{g L}^{-1}$  for  $\text{F}^-$ ,  $\text{BrO}_3^-$ ,  $\text{Cl}^-$ ,  $\text{NO}_2^-$ ,  $\text{Br}^-$ ,  $\text{NO}_3^-$  and  $\text{PO}_4^{3-}$ , respectively. LOD was calculated as:  $\text{LOD} = 3.3 \times s_0'$ , where  $s_0'$  is the standard deviation:  $s_0' = s_0/\sqrt{n}$ .  $s_0$  is the estimated standard deviation of  $m$  single results at or near zero concentration.

The limit of quantification (LOQ) were found to be 0.002, 0.01, 0.02, 0.01, 0.007, 0.002 and 0.01  $\text{mg L}^{-1}$ , respectively for the same anions. LOQ was calculated as:  $\text{LOQ} = kQ \times s_0'$ , where factor  $kQ$  value is 10 by IUPAC to RSD of 10 % and 5 or 6 to RSD value of 20% and 17% respectively.

Acknowledgments: This study has been financially supported by the Operational Programme "Science and education for smart growth" 2014-2020 of the European Union cofounded by the European Social Fund through the project BG05M2OP001-2.009-0015

**VALIDATION OF METHOD DETERMINATION OF CADMIUM IN DEPOSITED WASTES FROM BARITE MINE****VALIDATION DE LA DÉTERMINATION DE LA MÉTHODE DU CADMIUM DANS LES DÉCHETS DÉPOSÉS DE LA MINE DE BARITE**

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Cadmium is one of the most toxic in the heavy metal group whose threshold limit values is lowest in environmental regulatory requirements. The deposited wastes from the mining industry are a source of contamination of surrounding soils, groundwater and surface water and air. Methodology of extraction and measurement of cadmium in sterile dump waste from Tarnita-Suceava, Romania barite mine is validated in this study. The total heavy metal content in waste material and soils is established in previous studies. High content of arsenic, copper, lead and zinc were measured. Cadmium content ranges between 7-13 mg/kg in sterile dump and <0.5 mg/kg in surrounding soils. Analyzing such low concentrations requires multiple sets to confirm the results. An aqua regia extraction is applied to samples. Measurement is done with ICP-OES spectrometer. Analytical characteristics of the developed method limit of quantification, detection limits, working range, accuracy and precision were estimated.

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## GENERATIONS OF BIOMASS BASED FUELS

## GÉNÉRATIONS DE COMBUSTIBLES A BASE DE BIOMASSE

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### *Abstract*

The progressive depletion of fossil fuels and oil reserves together with the negative climatic impact of carbon dioxide emissions from fossil fuel burning have driven the research work in search of suitable fuel alternatives. The logical answer for a sustainable development is the development of technologies for biofuels production.

Biomass is considered environment-friendly source for renewable energy. Raw materials that can be used to produce biofuels are widely available worldwide and come from a large number of different sources and variety of forms. The concept is to constantly minimise the use of fuels based on petrochemicals by substitution of biomass based ones to meet the growing energy demand and reduce the impact of global warming.

In this paper three generations of biofuels according to raw material and technology used for their production are introduced and discussed.

## THE BIOREFINERY CONCEPT

### LE CONCEPT DE BIORAFFINERIE

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#### ***Abstract***

Human activities such as fossil fuel combustion and land-use change are indicated as main culprits responsible for the disturbance in the Earth's climate. The desired shift from fossil based carbon towards renewable carbon from biomass is the milestone for the development of biorefinery facilities. Biomass serves as versatile renewable feedstock – it can be derived from starch/sugar or oil seed crops, trees and grasses, agricultural and forest residuals, aquatics and microbes.

To achieve enhanced utilization of the biomass, new biomass based systems – biorefineries, are being developed. There, production, conversion and utilization of bioproducts are to be carried out efficiently in sustainability. In this way the use of different biomass species for various bioproduct applications – biofuels and biochemicals, has led to the consideration of simultaneous production of value-added co-products within the framework of a biorefinery.

In this paper different approaches to classify the biorefinery systems according to the defining criterion are described and discussed.

**CHEMICAL COMPOSITION OF DIFFERENT TYPES OF COMPOST**  
**COMPOSITION CHIMIQUE DE DIFFÉRENTS TYPES DE COMPOST**

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**Abstract:**

The purpose of current article is to present done investigation for chemical composition of three type's compost prepared on base of different row materials. For producing of two from the composts was applied "home" composting method under control of the conditions. The third one was produced in composting installation from "tunnel" type.

They was made studies for chemical composition for content of basic nutrients (N, P, K) and for content of some microelements and heavy metals (Ca, Mg, Na, Cu, Fe, Zn, Ni, Cd, Mn, Pb, Cr, As, Se, Sn, Bi, Sb). Also they are examined the parameters – pH and conductivity.

Obtained results clarified the chemical composition of the composts. On base of them are prepared adequate conclusions and recommendations.

## WHY ANALYSES OF GENETIC VARIATION IN NATURAL POPULATIONS OF MEDICINAL PLANT SPECIES ARE AN IMPORTANT COMPONENT OF THEIR FUTURE UTILIZATION?

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The chemical interaction between plants and their environment is mediated mainly by the biosynthesis of secondary metabolites, and often includes variation in their production levels. Specimens of same plant species growing under different environmental conditions exhibit significant differences in the synthesis of primary and secondary metabolites. In most cases, the natural populations of medicinal plants are extensively exploited due to their heavy demands. Thus, their long-term survival depends on the maintenance of sufficient genetic variability. Therefore, the knowledge and understanding of the patterns of genetic variation within and among populations of medicinal plants is essential for planning future management strategies for their conservation and sustainable utilization of their genetic resources, as well as for bioprospecting for new sources of value for pharmaceutical research projects. The aim of the present study is to present our case studies and modern trends in the genetic diversity assessments of medicinal plant species. Such studies, when performed hand in hand with metabolite profiling, would be quite helpful for identification of elite population of given species endowed with maximum content of active constituents. Low cost and easy access molecular approaches are also discussed herein.

**PRELIMINARY RESEARCHES WHICH MAKE THE DEVELOPMENT OF A SPECTROPHOTOMETRIC METHOD USED TO DETERMINE THE DYE CONTENT IN SMOKE GRENADES POSSIBLE.**

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The Bulgarian military forces need a method that gives them the possibility to find dye content in smoke grenades. Smoke grenades are widely used for military purposes such as creating smoke screens for camouflage and for signaling of aircraft. Preliminary researches had been made which make the development of a spectrophotometric method used to determine the dye content in smoke grenades possible.

The smoking mixture consists of dye, oxidant and sugars. The color of the smoke is determined by the dye that is in use. Mixing exactly weight components is the usual way for mixture production. However, analysis of the mixture is a must, in order for it to meet certain quality control requirements. What is needed is the creation of a method with good accuracy, precision and last, but not least a low cost.

This report presents the results of this spectrophotometric method for determination of dye content in a mixture for smoke grenades. The spectrums of the following dyes in DMSO that were studied: 1,4-dihydroxyanthraquinone (Solvent Orange 860), 1-(Methylamino)-9,10-anthracenedione (Solvent RED 111), 1,4-Bis((4-methylphenyl)amino)-9,10-anthracenedione (Solvent Green 3), 4-((1,5-Dihydro-3-methyl-5-oxo-1-phenyl-4H-pyrazol-4-ylidene)methyl)-2,4-dihydro-5-methyl-2-phenyl-3H-pyrazol-3-one (Solvent Yellow 93). The interferences were studied. The results showed that sugars and oxidant ( $\text{KClO}_3$ ) did not affect the absorbance of dye in the wave range 300-700 nm. Multipoint external standard calibration was applied. Beer's law was obeyed up to  $1 \times 10^{-4}$  mol/L dye with correlation coefficient 0.9999. Molar absorptivity of each dye was determined: Solvent Orange 860  $\epsilon = 7.4 \times 10^3$  L/mol.cm at  $\lambda = 475$  nm; (Solvent Yellow 93)  $\epsilon = 2.5 \times 10^4$  L/mol.cm at  $\lambda = 400$  nm; Solvent Green 3  $\epsilon = 9 \times 10^3$  L/mol.cm at  $\lambda = 645$  nm; Solvent RED 111  $\epsilon = 7 \times 10^3$  L/mol.cm at  $\lambda = 510$  nm.

The model samples that were used for the analysis contained dye from 46% to 54%,  $\text{KClO}_3$  and sugar in 1:1 mass ratio. The method showed good accuracy (recovery 98.6 - 103.6 %) and precision (RSD 3.8%). The developed method was applied in real samples obtained from a Bulgarian factory.

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**key words:** spectrophotometry, dye determination, smoke mixture, smoke grenade, analysis

## INVESTIGATION ON MULTIFUNCTIONAL ALLOY - TRITANIUM USED IN ORTHODONTICS

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In the past few years a multifunctional metal alloy-Tritanium is used in medicine, in orthopedics as outer layer for joint implants and recently Tritanium has been introduced on the market as an orthodontic archwire. The Tritanium archwire has three distinct force segments - anterior, bicuspid and posterior and could be useful in early stage of orthodontic treatment. During treatment, the archwires in the oral cavity, they can change their properties. The aim of our study is to characterize new type, multifunctional alloy - Tritanium used in orthodontics. For this purpose as-received tritanium archwire, with dimensions 0.016x0.022 inches<sup>2</sup> (0.40x0.55mm) was analyzed. The tests were made in the three distinct force segments. The samples were analyzed by: X-ray diffraction analysis (XRD), Scanning Electronic Microscopy (SEM), Energy Dispersive Spectroscopy (EDX), laser-induced plasma spectroscopy (LIPS) and Differential Scanning Calorimetry (DSC).

The investigated alloy is made mainly from the elements Ni and Ti. From EDX and LIPS analyses it is established that there are no significant chemical composition changes on the surface of the investigated archwires. The results obtained from DSC made in all segments of the Tritanium archwire in the temperature range from -50°C to +50°C shows that besides austenite to martensite transition, there is a presence of a rhombohedral intermediate phase (R phase), but with different temperature transition range for each investigated segment. These results obtained for as-received Tritanium archwires would be useful for further investigation of the archwires during orthodontic treatment.

Acknowledgment: This work was a part of a bilateral project between the Bulgarian Academy of Sciences and Estonian Academy of Science, Tallinn University of Technology

## EVALUATION OF THE MECHANICAL BEHAVIOR OF SHORT REINFORCED CONCRETE CORBELS STRENGTHENED BY BONDING OF CARBON FIBER FABRICS (CFRP). ANALYTICAL PROCEDURE FOR THE PREDICTION OF ULTIMATE LOAD

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The objective of this study is the proposition of an analytical procedure able to calculate the ultimate load capacity, to describe the evolution of the geometrical and physics parameters who governs the evolution of the resistance of a short reinforced concrete corbel strengthened by carbon fiber sheets (CFRP).

Modeling strategies are based on the use of damage mechanics associated with behavioral models for the constitutive materials (concrete, rebar, and CFRP).

The study focuses in particular on the influence of the type of carbon fiber fabrics, the orientation of the composite fabrics and the number of layers of carbon fiber fabrics.

Thus, different mechanisms have to be integrated into an analysis of the overall behavior of the structure, such as damage of the concrete, the elastic and elastoplastic behavior of reinforcing rebars or the behavior of carbon fiber fabrics and the adhesion failures at the CFRP-concrete interfaces. The phenomena are coupled and we will present how the different couplings will be taken into account.

The reinforcement by plating and coating of CFRP for the reinforced concrete structures considered is reproduced within the modeling by adding different types of carbon fiber fabrics with the specific characteristics of CFRP and changes in the parameters of the concrete model behavior, justified by experimentation and literature.

The relevance of the approach is finally highlighted by comparing the ultimate strength of the reinforced corbel with the experimental results.

**Keywords:** *strengthening, short console, mechanical behaviour, composite materials, reinforced concrete, ultimate load.*

## MICROSCOPIE ÉLECTRONIQUE ET LES OPORTUNITÉS QU'ELLE OFFRE À LA SCIENCE DES MATÉRIAUX MODERNE ET AUX NANOTECHNOLOGIES

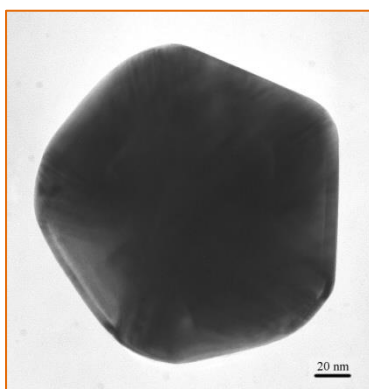
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Dans son essentiel, la microscopie électronique est une méthode de visualisation de la structure de la matière et, grâce à ses modes de diffraction et à ses analyseurs supplémentaires, elle fournit des informations sur les paramètres cristallographiques, la composition chimique et la présence de phases différentes. La capacité d'obtenir cette information est l'interaction physique complexe du faisceau d'électrons accélérés à grande vitesse avec le matériel étudié et l'enregistrement et l'analyse subséquents des rayonnements secondaires.

Historiquement, le premier prototype d'un microscope électronique a été créé par Max Knoll et Ernst Ruska en 1931, mais leur véritable reconnaissance n'est apparue qu'à la fin du siècle quand Ruska a reçu le prix Nobel en physique pour cette invention. Le fait n'est pas accidentel, car c'est le moment quand les nanomatériaux et les technologies attachées commencent à se développer, et la microscopie électronique s'est transformée dans une des méthodes indispensables pour leur étude - de la synthèse aux applications pratiques.



Nanoparticule d'or, images en modes de champ clair et de diffraction.



TEM JEOL JEM 2100, Le Laboratoire de microscopie électronique, L'Institut des Matériaux et Technologies Optiques "Académicien Jordan Malinovski", L'Académie Bulgare des Sciences

**SILICA BIOCOMPATIBLE HYBRID COATINGS****FOR WATER PURIFICATION**

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Recently a significant attention has been paid on the sol- gel method because of the possibility of obtaining new silica biocompatible hybrid materials with nanoscaled structure. These nanomaterials are excellent matrices for immobilization of different kind of biomolecules, which can be used for water purification.

In the present work the research results on sol- gel synthesis, structure and application of silica biocompatible hybrids have been described and discussed. The samples have been prepared at room temperature as coatings. The biocompatible hybrid materials are based on silica network with participation of two organic components – chitosan (CS) and hydroxyl ethyl methacrylate (HEMA), synthesized via sol - gel technique. The results based on FTIR and SEM analysis presented formation of homogeneous structures, which exhibit improve reactivity.

The ability of obtained hybrid materials to form biofilm of immobilized cells is established via deposition as coating on SiO<sub>2</sub> substrates using dip coating technique. Based on the obtained results is evaluated, that immobilized cells are evenly distributed all over the coating surface. Furthermore, improve quantity of cells due to biocompatibility and reactivity possesses the silica biocompatible hybrid coat In the present work the research results on sol- gel synthesis and structure of hybrid nanocomposites have been described and discussed. The samples have been prepared at room temperature as flakes.

## CHEMICAL PROCESSING OF POWDERS FOR METAL (AL & TI) MATRIX COMPOSITES USED IN AERONAUTICAL APPLICATIONS

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### Abstract

Metal (Al & Ti) matrix composites have been fabricated after thermomechanical doping by powder metallurgy route using biaxial cold compaction.  $\text{Al}_2\text{O}_3$  has been added in AA7075 as a major reinforcement. In another process, chemical treatment was carried out by using boron (B) and  $\text{TiO}_2$  to obtain  $\text{TiB}_2$  ceramic component. It means that a thermos-mechanical doping has been done to obtain  $\text{TiB}_2$  that will be used later in the Al-Cu-Ti matrix for increasing the micro-hardness capacity and wear resistance under the service conditions.

In this work, a special design of two different metal matrix composites have been carried out by using hard ceramics oxides. At the first composite design,  $\text{Al}_2\text{O}_3$  has been added in AA7075 as a major reinforcement with a special doping process. At the second composite design, a chemical treatment was carried out by using boron (B) and  $\text{TiO}_2$  to obtain  $\text{TiB}_2$  ceramic component at high temperature. It means that a thermo-mechanical doping has been done that will be used later in the Al-Cu-Ti matrix for increasing the micro-hardness capacity and wear resistance under the service conditions. Critical for the reaction is the time spend for milling and doping. Longer milling time will result in finer particle size and elimination of possible agglomerations.

**Keywords:** Composites, reinforcement, powder metallurgy, SEM.

## DESIGN OF LOW COST COMPOSITES FROM RECYCLED ALUMINIUM + COPPER FOR TRIBOLOGICAL APPLICATIONS

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### Abstract

A special aluminium-copper metal-matrix composite (ACMMCs) obtained from the fresh scrap chips of AA7075 and pure electrolytic copper were designed through combined method of powder metallurgy and sinter-forging. In the frame of this common project, novel composites have been developed from scrap copper + aluminium AA7075 powder reinforced with Nb<sub>2</sub>Al and SiC particles as main reinforcements in an economic way. An Al-Cu matrix was doped with ZnO after the ball milling with two basic reinforcements (Nb<sub>2</sub>Al– SiC, etc.) was carried out during the 4 hours. A basic composition was prepared depending on the doping percentage of ZnO as 30wt%.

Low cost manufacturing of these composites have been successfully managed through the combined method of sinter + forging. Microstructural analysis has shown that a good bonding at interface of matrix-reinforcement essentially in the specimens manufactured with combined process sinter + forging; a tough and sound microstructure was obtained without porosity. Mechanical behaviours of these specimens produced with sinter + forging process are better than the specimens produced with only sintering processes.

**Keywords:** Composites, reinforcement, recycled aluminium, SEM, tribological applications.

## GARANTIE ÉCOLOGIQUE POUR INSTALLATIONS DE RECYCLAGE DES BATTERIES LITHIUM-ION

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Au cours des deux dernières décennies, les exigences techniques pour les batteries sur le marché des entrepôts d'énergie portables ont augmenté et sont devenues de plus en plus complexes. Les batteries Lithium-ion (BLI) sont considérées comme la source d'énergie la plus prometteuse pour les applications mobiles et industrielles car elles présentent plusieurs avantages par rapport aux autres types de batterie.

Au niveau européen, la directive „Battery 2006/66 EG“ est le cadre juridique pour le recyclage des batteries. La directive sur les batteries définit les différents types de batteries et les systèmes de collecte et les quotas de marché, les interdictions des matériaux utilisés dans les batteries et les quotas de recyclage pour les différents types de batteries.

Le recyclage des batteries Lithium-ion jouera un rôle de plus en plus important dans le futur, car la proportion d'utilisation des batteries Lithium-ion continuera à augmenter dans les années à venir. L'objectif des processus de recyclage est de séparer les constituants en différentes fractions pour les réintroduire en production, afin de minimiser le type et la quantité de déchets générés, d'immobiliser et de neutraliser les substances dangereuses. La récupération des métaux (Ni, Co, Cu, etc.) est l'une des principales incitations au recyclage des batteries.

L'effort réel consiste à produire des produits utilisables, à minimiser le type et la quantité de déchets générés, à réformer les substances dangereuses et à le faire de manière écologique et économique environnementale. La récupération des métaux (Ni, Co, Cu, etc.) est l'une des principales incitations au recyclage des batteries.

La quantité élevée d'énergie stockée, les composants toxiques et le risque potentiel d'incendie sont les raisons pour lesquelles les batteries lithium-ion créent plusieurs problèmes qui compliquent le processus de recyclage.

On connaît différentes techniques qui peuvent être utilisées pour le recyclage des batteries Lithium-ion portables et industrielles. Ils peuvent être divisés en trois catégories principales : processus mécaniques, processus pyrométallurgiques et processus hydrométallurgiques (ou combinaison).

Dans la présente étude une méthode de prétraitement mécanique pour le recyclage des batteries au lithium-ion mobiles et industriels a été développée. Cette méthode peut être étendue en incluant des phases supplémentaires impliquant des méthodes pyro-hydrométallurgiques.

**SYNTHESIS OF  $\text{ZnTeO}_3$  BY CRYSTALLIZATION OF GLASSES IN THE** **$\text{TeO}_2$  -  $\text{Bi}_2\text{O}_3$  -  $\text{ZnO}$  -  $\text{Nb}_2\text{O}_5$  SYSTEM****SYNTHÈSE DE  $\text{ZnTeO}_3$  PAR CRISTALLISATION DE VERRES DANS LE SYSTÈME** **$\text{TeO}_2$  -  $\text{Bi}_2\text{O}_3$  -  $\text{ZnO}$  -  $\text{Nb}_2\text{O}_5$** 

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The aim of the present work is to verify the synthesis of zinc tellurite by crystallization from glasses in the  $\text{TeO}_2$  -  $\text{Bi}_2\text{O}_3$  -  $\text{ZnO}$  -  $\text{Nb}_2\text{O}_5$  system. The investigation covers  $\text{TeO}_2$  - based glasses containing  $\text{Nb}_2\text{O}_5$  and  $\text{Bi}_2\text{O}_3$  up to 10 mol % and  $\text{ZnO}$  from 5 to 10 mol %. Several glasses were selected and heat treated at 500°C for 6h.

The obtained glasses were transparent and bright yellow colored. The thermal stability of the samples was determined by DTA using the difference  $\Delta T$  between exothermic peak of crystallization ( $T_x$ ) and that for glass transition temperature  $T_g$  ( $\Delta T = 50 \div 115^\circ\text{C}$ ). The XRD analysis detected formation of two crystalline phases  $\text{ZnTeO}_3$  and  $\text{ZnO}$ .

Additional information for the formation of  $\text{ZnTeO}_3$  crystal phase was obtained by infrared spectroscopy (IR). There is a difference in the IR spectra of binary sample 50 $\text{TeO}_2$ .50 $\text{ZnO}$  ( $\text{ZnTeO}_3$ ) and multicomponent ones. The analysis of spectra shows that the glass network consists mainly of  $\text{TeO}_4$  (tbp) units, while  $\text{TeO}_3$  (tp) are the main structural units in the crystalline  $\text{ZnTeO}_3$  phase.

The microstructure of selected crystallized samples was examined by SEM analysis. The preliminary microprobe chemical composition analysis showed presence of  $\text{ZnTeO}_3$  phase that is in agreement with the XRD results.

## DESIGN, EXAMINATION AND EVALUATION OF REINFORCED HYDROXYAPATITE FOR BIO-APPLICATIONS

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### Abstract

Hydroxyapatite ( $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ ; HA) is the mineral matrix of the bone. It is the most widely studied bioactive and biocompatible material. For improving the mechanical properties six reinforcements were used (B/BN, Mg/MgO, Zn/ZnO). For each reinforcement a series of specimens with varying percentage was made (3, 6, 9 wt% B/BN and 2, 4, 6 wt% for Mg/MgO, Zn/ZnO) examined and evaluated. For the sintering conditions a heating rate of 3-5 °C/min at 1200 °C for 4 hours was used. Various sintering aids were considered and used including  $\text{CaF}_2$ , PVA, Ytria-stabilized zirconia (YSZ),  $\text{B}_2\text{O}_3$  and  $\text{H}_3\text{BO}_3$ .

These reinforcements were selected because they are naturally seen in HAP. In combination with HAP they are osteoinductive and osteoconductive. All of the reinforcements are present in the human body in the form of vitamins or trace elements. In addition, when they are added to the bone mineral matrix, they act as structural and mechanical reinforcement. The examined compositions can be used as bone cement fillers or probably as implant coating.

**Keywords:** HAP, reinforcement, bio-application, ceramic.

## REDUCED GRAPHENE OXIDE AND COLLAGEN COMPOSITES

### COMPOSITES D'OXYDE DE GRAPHÈNE REDUIT ET DE COLLAGÈNE

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Antimicrobial activity is desirable characteristic of collagen bio-materials. One of the simplest and most efficient ways to achieve it is to create composites that include antimicrobial agents, usually nanoparticles.

The Hammer method was used to synthesize reduced graphene oxide by chemical exfoliation of purified natural graphite powder. It was proved that the obtained reduced grafted oxide is a multi-layer sheet of high purity and a number of layers up to five by means of X-ray analysis, CEM and TEM.

We obtained porous collagen/RGO composites with an open and interconnected porous structure with varying weight ratios. The studied collagen/RGO composites show a pronounced antimicrobial effect against Gram+ bacteria and the lack of Gram-bacteria as well as the antifungal effect. With increasing RGO content in collagen composites, antibacterial and antifungal activity increases.

Collagen bio-materials are used for tissue engineering and regenerative medicine.

**SYNTHESIS OF COMPOSITES OF SUPERCONDUCTING BSCCO CERAMIC WITH  
FERROMAGNETIC MANGANITE PHASES****SYNTHESE DE COMPOSITES DE SUPERCONDUCTEUR DE CERCOT DE BSCCO  
AVEC DES PHASES DE MANGANITE FERROMAGNÉTIQUES**

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Two different methods – a solid state reaction and low temperature sol-gel method were used to obtain bulk composites based on superconducting BSCCO ceramics and manganite phase.

The microstructure of obtained samples was studied by X-ray diffraction, scanning electron microscopy (SEM) and the method of energy disperses spectroscopy (EDX). The hysteresis curves and the magnetization curves of the composites were constructed by using the PPMS - Physical Properties Measurement System.

It was proved that the obtained samples combine the properties of the two starting components - superconductivity at temperatures below the critical (62-86K) and ferromagnetism under Curie temperatures (330-350K). It was found the positive influence of lanthanum manganite on the critical current density  $J_c$  of the resulting composite which significantly exceeds the values for the unalloyed superconductive BPSCCO ceramics.

This composite is a potential candidate as multifunctional material for applications in microelectronics.

## OXYDATION ANODIQUE D'ALLIAGES DE TITANE DANS DES ELECTROLYTES CONTENANT DU FLUOR

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### RESUME :

Dans le présent article, la croissance du film anodique sur les alliages de Titane (Ti) dans des électrolytes d'éthylène glycol contenant de l'eau et du Fluor ont été étudiés en utilisant des techniques d'analyse électrochimique et de surface. Les courbes de potentiel de courant en régime permanent et les spectres d'impédance électrochimique ont été établis en fonction du potentiel et de la concentration en Fluor indiquant deux voies de réaction parallèles - croissance /dissolution du film et la dissolution du Titane à travers le film. La composition chimique des films anodiques a été estimée par spectroscopie photo-électronique des rayons X (XPS) . Les analyses XPS ont révélé la présence d'un oxyde en rapport non stœchiométrique contenant principalement du  $Ti^{4+}$  et une certaine quantité de  $Ti^{3+}$ , avec un certain degré d'hydroxylation. Mo et Al se sont révélés être riches en oxydes, avec une nette évidence d'oxydation et de dissolution transpassive de Mo. Un modèle cinétique du processus est proposé et ses paramètres sont estimés par comparaison quantitative avec le potentiel et les données EIS. Les caractéristiques principales des données XPS, à savoir l'épaisseur du film, la concentration de Ti (III) et du Fluor, en fonction du potentiel sont également reproduites par le modèle.

### MOTS CLES :

- \*Alliage de Titane
- \* Eau, Fluor, électrolytes d'Éthylène Glycol
- \* Oxydation anodique
- \* Spectroscopie à impédance électrochimique

**PRODUCTION DES MATERIAUX MICRO- ET NANOSTRUCTURES DE POLYCARBONATE RECYCLE PAR LA METHODE D'ELECTRO-FILAGE**

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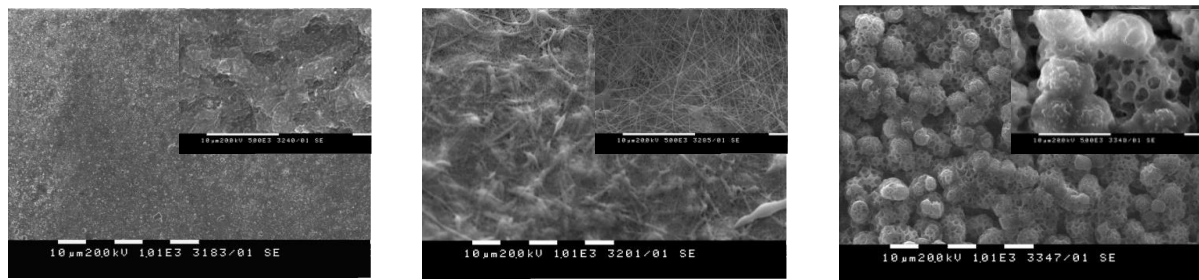
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Les polymères utilisés dans la vie quotidienne de la société ensemble avec d'autres matériaux non dégradables utilisés par l'homme se transforment en déchets qui s'accumulent de plus en plus dans la nature et provoquent des effets négatifs. Les changements climatiques globales dans les dernières années ont mené vers des décisions et des mesures politiques et économiques pour diminuer les quantités de ces déchets et de stimuler la réutilisation de ces produits au niveau de la recherche et de la production.



Dans ce travail est démontré la possibilité d'utiliser le polycarbonate, l'un des polymères, les plus répandus et qui est déchets de la construction, pour produire des systèmes micro- et nanostructurés avec un potentiel d'application dans des domaines différents – protection des surfaces, filtrage etc. Des conditions pour la dissolution du polymère dans un solvant spécifique, composé de TCM (trichlorométhane) et TCA (acide trichloroacétique) en proportions divers sont établies et la solution est utilisée comme précurseur d'effectuer l'électro-filage. Comme cette méthode est sensible aux propriétés de la solution - la viscosité et la conductivité, mais aussi aux paramètres expérimentales - la tension appliquée, la distance entre la source de la solution et le collecteur, en les changeant, trois différents types de morphologies sont reçues: couches minces, nano fibres et microsphères poreuses. Pour étudier la structure de la surface de ces objets nous avons utilisé un microscope électronique à balayage digitalisé Philips 515 et un profilomètre optique Zeta-20. Les propriétés des couches et des fibres reçues de protéger un substrat (de papier et de tissu) contre l'attaque chimique de base et d'acide sont discutées. Fig. 1. Images de microscopie électronique à balayage des trois types de morphologie de polycarbonate recyclé par la méthode d'électro-filage.

**PHYSICO-CHEMICAL CHARACTERIZATION OF NEW TELLURIUM BASED  
CHALCOGENIDE MATERIALS****PROPIETES PHYSISCO-CHIMIQUES DE NOUVELS MATERIAUX  
CHALCOGENIDES A LA BASE DE TELLURIUM**

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Bulk samples of the Ge-Te-Cu system were synthesized by melt-quenched technique into a mixture of water and ice to obtain amorphous materials. The synthesis was carried out at a constant heating rate of 3°C/min to a final temperature of 1100°C. Some physico-chemical properties of the synthesized bulk samples were defined as the average coordinate number, density, compactness, molar volume and free volume and their dependence on the composition was investigated. The density of the bulk samples was determined by the pycnometric method. With an increase of Cu content, the density undergoes various changes, most likely due to the gradual substitution of tellurium atoms with copper, leading to structural changes in the samples. The dependencies of the compactness, molar volume and free volume of the bulk sample composition follow that of density. According to theory of Thorpe for transitions in the structure of covalent chalcogenide glasses at an average coordination number 2,4 in the system GeTe<sub>4</sub>-Cu at  $Z_{\text{glass}}=2,46$ , corresponding to a composition with 10mol% Cu, occurs a change in the structure of the bulk samples, which leads to extreme in the physico-chemical properties.