Phytochemical characterization and anti-oxidant activity of the prune by-product

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Abstract

Recovering of food waste is one of the main challenges and a global goal given the pollution and generation of waste [1]. This study aims to valorize the by-products of Prunus genus in order to determine the chemical composition and the biological potential of Prunus domestica L collected in France. The extracts have been carried out by successive extractions with solvents of increasing polarity (Dichloromethane, Ethyl acetate and methanol) and directly extraction with an ethanol solvent. GC-MS (Gas Chromatography – mass spectrometry) was used to investigate chemical composition. Furthermore, the measurement of total polyphenols content was determined by the Folin Ciocalteu assay. In addition, the antioxidant power of plant extracts was evaluated by the DPPH (1, 1-diphenyl-2-2-picrylhydrazyl free radical) assay. Results show that the methanol extract at 50mg/L concentration had the highest antioxidant activity with a 22.8% of inhibition. It also had the highest phenolic content with 8.9 mg of gallic acid equivalents (GAE)/g of dry weight. GC-MS analysis revealed some volatile compounds before and after derivatization. Among the latter 5-hydroxymethylfurfural, benzaldehyde, 9,12-octadecanoic acid ethyl ester, 1-Hexacosanol, pulegone, D-Glucose, D-Allofuranose, palmitic acid, linoleic acid, D-(+)-Xylose and benzoic acid. These compounds, identified as hydrocarbons, alcohols, alkanes and esters, represent the main classes of volatile substances in plums and explain their discreet aroma.

Highly efficient red-emitting hetero-bimetallic complexes and their application in light emitting electrochemical cells

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Among phosphorescent organometallic compounds, cyclometalated Ir^{III} complexes are the most promising emitters for application in light-emitting devices, such as organic light emitting diodes (OLEDs) and electrochemical cells (LECs). Although mononuclear compounds are leading compounds in the field, multimetallic counterparts are much less explored, in spite of the several advantages provided by the presence of additional heavy metal centers. We herein report on a novel class of phosphorescent cationic hetero-bimetallic Ir^{III}/M^{I} complexes, where M^{I} = Cu^{I} and Au^{I}. The complexes display vibrant red emission with an enhanced radiative rate constant and improved photoluminescence quantum yield when compared to their mononuclear congeners. In particular, some of these derivatives are amongst the most efficient cationic dinuclear red-emitting Ir^{III} complexes with a \(\Phi_{PL}\) as high as 72-77% in degassed dichloromethane solution and lifetime in the range 1.8 \(\mu\)s. Remarkably, their use as electrolative materials in single-layer LEC devices is demonstrated achieving fast turn-on time, brightness up to 1100 cd m^{-2} and external quantum efficiency up to 6%.

Pines species are worldwide cultivated specially in both France and Tunisia. The different part of pines (needle, cone, bark and resin) contains various medicinal values and were traditionally used in rheumatism treatment and as antiseptic agent. The current study was aimed to determine the chemical composition of Pinaceae’s extracts obtained by maceration using two solvents (cyclohexane and ethyl acetate). The chemical composition were performed by GC-MS (Gas Chromatography Mass Spectrometry) method. The GC-MS results were led to identify more than 10 chemicals compositions such as a isopinocarveol; Bicyclo[3.1.1]hept-3-en-2-one, 4,6,6-trimethyl-; Bicyclo[3.1.1]hept-2-ene-2-carboxaldehyde, 6,6dimethyl-; Bicyclo[2.2.1]heptane-2,5-diol, 1,7,7-trimethyl-, (2-endo,5-exo)-; Longifolene...

Since expansion of antimicrobials, many antibiotic-resistant bacteria have developed and force scientists to redouble their inventiveness. For the past decade, carbon monoxide (CO) has been one of the alternatives. This molecule identified as inhibiting bacterial proliferation seems to be a good candidate. However, given the furtive dangerousness of CO gas, it is necessary to use it in a roundabout way. In this perspective of controlled release, PhotoCORMs (photochemically CO-releasing molecules) can be used.

PhotoCORMs based on tricarbonyl-rhenium (I) developed in the team can be used clinically due to their biocompatibility, their generation of CO, production of reactive oxygen singlet, while following the reaction by a color change. However, it is important to improve water solubility of these charged but hydrophobic complexes. In this context, fixing the complexes on a water-soluble and biocompatible platform such as cellulose nanocrystals (CNCs) is a good option. The negative surface of CNCs allows both to adsorb active and recognition molecules directly or via functionalized cyclodextrins.

In this context we sought to develop and characterize the photoreactivity of two systems in order to study their antibacterial properties in the future. A first system with a rhenium complex adsorbed on CNCs and another system composed by a rhenium complex with an adamantyl end encapsulated in a charged cyclodextrin, which is adsorbed on the CNCs (figure 1).

**Figure 1**: Schematic representation of prepared nanomaterials

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Effect of extraction techniques on phenolic content and biological activities of *Scorzonera undulata* organs

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The genus *Scorzonera* contains various medicinal values [1]. Species belong to this genus were traditionally used as drugs or in food. In this study, maceration, and ultrasonic-assisted extraction were used to extract phenolic compounds from leaves, roots, and flowers of *Scorzonera undulata*. Total phenolic content was measured by the Folin–Ciocalteu assay. Furthermore, the chemical composition of *S. undulata* extract was also investigated by the LC-MS method using phenolic acid and flavonoid standards. The variation of the extraction methods induced a variation in the real potentialities of the three organs in terms of bioactive molecules. However, the aerial part of *Scorzonera undulata* (leaves and flowers) showed, in general, the highest phenolic contents. The phytochemical analysis by LC-MS revealed a high content of quinic acid, chlorogenic acid. The antioxidant activity and the antidiabetic activity of each extract were evaluated by *in vitro* tests. The DPPH test showed that the aerial part of the plant has a higher antioxidant activity compared to the root (25.06% at 50 mg/L for the leaf ethanolic extract obtained by ultrasound extraction). In effect the evaluation of the anti-enzymatic activity (anti α-amylase) showed that the aqueous extract of the flowers prepared by ultrasound has the highest activity (26.23% at 50 mg/L).

PAH photo-reactivity with water clusters and water ice: structures, energetics and spectra from FTIR experiments and a multi-method theoretical study

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The interaction of polycyclic aromatic hydrocarbons (PAHs) with water is of paramount importance in atmospheric and astrophysical context. PAHs are believed to be a significant reservoir of carbon in the interstellar medium (ISM) and have been proposed to be the carriers of the Aromatic Interstellar Bands (AIBs), a set of infrared emission bands observed in many regions of the ISM. In dense and cold environments, PAHs are likely to condense onto or integrate into water ice mantles covering dust grains and to contribute to the complex grain chemistry. Understanding the role of water in the photo-processes involving adsorbed PAHs is therefore a key issue in astrochemistry.

Our joint theoretical (classical MD / force field simulations and SCC-DFTB calculations) and experimental (matrix isolation FTIR spectroscopy) studies have revealed the key role of mutual orientation in PAH-water interactions and reactivity. Both PAH (planar or not), and ice (amorphous or crystalline) structures play a role in the formation of PAH-water complexes and on their photo-reactivity. Various results from this wide-ranging study and their astrophysical implications will be presented:

- the role of water clusters and of ice structure on the photo-reactions of PAHs [1]
- the relationship between the number of interacting dangling OH (dOH) bonds of ice and PAH vertical ionization potential (VIP) variation [2].
- how the surface of amorphous solid water (ASW) is perturbed by the adsorption of PAHs, leading to a redshift and a broadening of the dOH ice spectral feature.
- the effect of the PAH structure by comparing coronene and corannulene photoreactivity with water [3].
- the description of PAH-ice interaction in the ground electronic state at low temperature, providing the binding energies and barrier heights necessary to the on-going improvement of astrochemical models.

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New Chromone-2-Carboxamides Based Analogs of Pseudomonas aeruginosa Quorum Sensing Signal as New Anti-Biofilm Agents

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Biofilm formation is considered a major cause of therapeutic failure because bacteria in biofilms have higher protection against antimicrobials [1]. Thus, biofilm-related infections are extremely challenging to treat and pose major concerns for public health, along with huge economic impacts. Pseudomonas aeruginosais, in particular, is a “critical priority” pathogen, responsible for severe infections, especially in cystic fibrosis patients because of its capacity to form resistant biofilms [2]. Therefore, new therapeutic approaches are needed to complete the pipeline of molecules offering new targets and modes of action. Biofilm formation is mainly controlled by Quorum Sensing (QS), a communication system based on signaling molecules [3]. In the present study, we employed a molecular docking approach (Autodock Vina) to assess two series of chromones-based compounds as possible ligands for PqsR, a LuxR-type receptor. Most compounds showed good predicted affinities for PqsR, higher than the PQS native ligand. Encouraged by these docking results, we synthesized a library of 34 direct and 25 retro chromone carboxamides using two optimized routes from 2-chromone carboxylic acid as starting material for both series. We evaluated the synthesized carboxamides for their ability to inhibit the biofilm formation of P. aeruginosain vitro. Overall, results showed several chromone 2-carboxamides of the retro series are potent inhibitors of the formation of P. aeruginosabiofilms (16/25 compound with % inhibition ≥ 50% at 50 µM), without cytotoxicity on Vero cells (IC50 > 1.0 mM) [4]. The 2,4-dinitro-N-(4-oxo-4H-chromen-2-yl) benzamide (6n) was the most promising antibiofilm compound, with potential for hit to lead optimization.

Chimie click in situ pour la formation de nouveaux inhibiteurs d’InhA de *Mycobacterium tuberculosis*

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En 2020, la tuberculose, causée par *Mycobacterium tuberculosis*, représentait la 13\ème cause de mortalité dans le monde et la seconde causée par une maladie infectieuse derrière la COVID-19. En 2020, ce sont 9.9 millions de personnes qui ont contracté la maladie dont 1.1 million d’enfants. Au cours des 60 dernières années, aucun nouveau médicament antituberculeux n’a été approuvé par la FDA aux États-Unis pour le traitement de la tuberculose multi-résistante, à l’exception de la bédaquiline, du délamanide et du prétomanide, pour lesquels des résistances sont déjà émergentes. C’est pourquoi il est nécessaire de découvrir de nouvelles drogues pour lutter efficacement contre la tuberculose. Les enzymes appartenant au système « fatty acid synthase » de type II (FAS-II), sont nécessaires à la biosynthèse des acides mycoliques, des composants de la paroi mycobactérienne essentiels à la survie des bactéries. Parmi ces enzymes, InhA, une énoyl-ACP réductase, permet la réduction des acides gras trans-2-enoyl-ACP. L’objectif principal du projet est de développer des outils qui permettent d’identifier de nouveaux inhibiteurs de cette protéine.

La chimie combinatoire propose différentes stratégies pour la production de molécules en grand nombre et permet l’identification de nouveaux « hits » en chimie médicinale. Parmi ces stratégies, la synthèse guidée par la cible sous contrôle cinétique (KTGS) est une alternative, la protéine-cible va participer à la synthèse de ses propres ligands. Cette synthèse *in situ* permet de relier les fragments réactifs entre eux d’une manière intermoléculaire et irréversible par confinement, via la protéine-cible, pour générer des molécules plus affines pour la cible. L’objectif du projet est de mettre en application la chimie click *in situ* intermoléculaire à l’aide de InhA par différents fragments possédant soit une fonction azide, soit une fonction alcyne. Un nouveau concept novateur est également étudié, la chimie click *in situ* intramoléculaire afin de macrocycliser une molécule acyclique portant un azoture et un alcyne. Dans cette approche basée sur des considérations stériques, nous prévoyons qu’un précurseur acyclique atteindrait la poche protéique plus facilement que son homologue macrocyclique. Une fois logé à l’intérieur de la poche, le confinement devrait favoriser la proximité des réactifs et faciliter la macrocyclisation intramoléculaire pour synthétiser *in situ* un ligand avec des propriétés de liaison plus fortes.

Grapes (*Vitis vinifera L.*) are one of the most cultivated fruits in the world with an annual production of 58 million tons in 2012 (Yammani, 2016) and the amount is increasing through time. About 80% of the production are used for wine making. The main residues are formed after the pressing stage: pomace and lees.

Much research has shown the great potential that wine by-products has since they are rich with bioactive compounds and their effective role in curing a lot of disease. We can mention the activities that we could find such as antioxidant, anti-diabetic, and anti-Alzheimer activity.

The pomace generated by pressing give a quantity estimated by 10 million tons per year and it is used as a soil fertilizer also as a food supplement since it is rich in phenolic compounds.

Our aim in this research is to find new eco-friendly way in order to extract the bioactive molecules like subcritical water and pulsed electric fields.

In this research we are comparing two types of wine Malbec and Cabernet Sauvignon, we are looking for to determine the chemical composition and chromatographic profiles of extracts using the HPLC technique.

The results that we have that Malbec contains the highest quantity of phenolic compounds: 65 mg EqACG/g MS.

References


Sami Yammini, « Extraction des sous-produits de la vigne du vin par eau sous-critique et purification des extraits par procédés membranaires ». p. 5.
Kinetics of palm by-product fermentation using Kombucha starter culture

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The date palm is the main wealth of southern Tunisian. It offers a wide range of agricultural by-products consisting mainly of cellulose, hemicellulose, lignin and sugar. This waste could be used in many industrial and biological processes. This work aims to study the fermentation of palm by-products. Fermentation was done following the Kombucha production procedure.

The crushed by-product left to infuse for 15 min at 80°C. After cooling, the infusion is inoculated with 20 g of SCOBY culture and fermentation was carried out at 25 °C for 15 days. All fermentations were performed in duplicate. The final biofilm was weighed (14g). The pH values of the samples were measured using an electronic pH meter. The results showed that the pH value goes from 6.7 to 2.7. This is due to the increase in acidity by organic acids derived from bacterial metabolites. HPLC analyzes were used to determine fermentation kinetics.
Evaluation of the chemical and biological properties of natural active extracts of *Burkholderia Sp*

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*Burkholderia sp* is a new Gram-negative soil bacteria discovered and isolated for the first time from a French agricultural soils generally used for wheat and barley cultivation. This genus play an essential role in defining and maintaining soil equilibrium by promoting plant growth (fix nitrogen, and degrade pollutants) 1. The objective of the current study was to determinate the chemical composition of *Burkholderia sp* by HPLC-DAD, GC-MS without and with derivatization. In addition, biological activities (anti-inflammatory, anti-acetylcholinesterase and anti-xanthine oxidase) were studied for *Burkholderia* extracts using organic solvents of increasing polarity: dichloromethane, ethyl acetate, butanol, and water. This bacteria demonstrated different volatiles profiles coming from varieties of compound classes such as alcohols, diketopiperazines, hydrocarbons, aromatic compounds and esters. Therefore, GC-MS results showed that 29 compounds were identified before derivatization such as octanoic acid, 2,5-piperazinedione, 3-benzyl-6-isopropyl-, 2,4-di-tert-butylphenol and pyrrolo[1,2-a]pyrazine-1,4-dione, hexahydro- and 13 additional ones after derivatization such as palmitic acid, glycerol and 2,3-butanediol. Furthermore, the obtained results of all the biological activities that have already been mentioned showed generally moderate to low inhibition percentage.

Wine by-product analysis, chemical and biological extraction and analysis

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Grapes are one of the most world-grown, and winegrowing generates significant amounts of by-products. Wine wastes are known to contain phenolic compounds such as polyphenols but also sugars. Several studies have demonstrated the antioxidant, biological and anti-tumour activities of these molecules present in red wine in particular. We studied two grapes varieties: Malbec y Cabernet-Sauvignon. Our work is aimed at extracting, dosing and analysing polyphenols from several wine waste.

A determination of total polyphenols was made by the reagent of Folin Ciocalteu, and a determination of sugars by the 2-hydroxy-3,5-dinitrobenzoïque (DNS). Furthermore, various activities were tested such as anti-alpha glucosidase activity, an enzyme involved in diabetic disease; the antioxidant activity was tested too (1,1diphenyl-2-picrylhydrazyl (DPPH) test) or the study of anti-inflammatory activity. Finally, cytotoxicity tests were made on specific cell line (Raw).

The different assays and tests showed us that the samples from Malbec have the most significant results. For the total polyphenols content of tested extracts, Malbec extracted with ethanol at 250 bar and 60°C has the highest content with an average of 55.5 mg eq AG/g extract. Malbec extracted with ethanol at 700 bar and 40°C has the most significantly inhibits of alpha-glucosidase at a concentration of 50 and 5 mg/L in the diabetes test; we obtained 83,04% and 15,55% respectively.

These tests allowed us to highlight the different properties of wine waste through its various activities. On the two grape varieties studied, Malbec is the most concentrated in polyphenols and has the best anti alpha-glucosidase activity. Further dosages and activities are planned and will have to be made in order to continue the research with the aim of recovering this waste in the pharmaceutical field. It is also about finding the eco-friendliest extraction method in the fight for the environment.

Key words: wine wastes, polyphenols, antioxidant, dosing, extraction, pharmaceutical field, by-products, analysis, chemical composition, biological activities.


These.fr. [https://www.theses.fr/2015COMP2249.pdf](https://www.theses.fr/2015COMP2249.pdf)
Control of helix screw-sense preference in dynamic oligourea foldamers of meso symmetry built from chiral monomeric units

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The screw sense preference of helical foldamers, like their biological counterparts (e.g. peptide and DNA), mostly depends on the chirality of their monomeric units. For example, N,N'-linked oligourea foldamers composed of enantiopure chiral ethylene diamine units in (S)-configuration fold into right handed (P-) helices stabilized by a network of intramolecular hydrogen bonds. Sequence-defined chiral helical foldamers which adopt a well-defined and predictable conformation have found widespread interest with potential applications ranging from biology to catalysis. However, to mimic the dynamic character of proteins for instance conformational change via allosteric regulation, more dynamic systems that could orchestrate function based not only on conformation but also on conformational changes induced by external stimuli are also desirable. Recently, studies of dynamic foldamers built from achiral (or meso) units have shown that properties such as helix handedness and conformation can be altered by environment. However, cognate dynamic foldamers solely composed of chiral units, thus similar to biopolymers, have not been studied yet.

Herein, we present structural analysis and control of helix screw sense preference of meso and near-meso oligourea foldamers built from chiral units. X-ray diffraction analysis of a meso oligourea revealed the presence of partially folded P- and M-helices in a racemic crystal. Breaking the symmetry by substituting one of the two identical terminal groups (extremities) convert meso into near-meso structure and induced a preferred helix-screw sense. The folding propensities of near-meso compounds may be modulated by using various end-group combinations. Solution phase analysis using CD and NMR are consistent with this extremities-dependent folding behaviour. Binding of achiral hydrogen bond acceptors to near-meso compounds reversed the direction of the intramolecular hydrogen bond network and resulted in helix handedness inversion. Moreover, this inversion could be used to trigger the catalytic activity of a foldamer. It is noteworthy that this substrate-induced conformational change and “triggerability” of a dynamic foldamer catalyst solely consisting of chiral units is highly reminiscent of induced-fit in enzymes.

GAFFEx.org, a platform to make “negative results” visible

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Despite the fact that most researchers are seeing in “negative results” useful data, many of those are sleeping in lab drawers, and their non-publication seems to be a growing phenomenon¹! Considering, “failed experiments” unavailability to the scientific community, is a signature that several barriers need to be overcome to leverage their publication², a dedicated place to promote the publication of “negative results”²(2, 3), GAFFEx.org is being developed. Designed to fit this specific type of scientific material, GAFFEx is trying to answer the following questions: what can we consider as “negative results”? Why should we publish them? Why is this type of results so difficult to share? Which type of incentives will help the publication and/or review?

We will discuss these questions and a team of volunteers, members of the non-profit organization Science Unchained, will present the solution they are preparing to fit researchers’ constraints when publishing their negative results. A call for volunteers will be opened to participate in testing and improving the platform before a first release by the end of 2022.

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Biographies

Gilmary Gallon is a Senior Associate Researcher at Medincell, where he is working on physicochemical drug properties. In 2017, he started the development of GAFFEX to improve the publication of scientific failing experiments, also called “negative results”. In 2019, he obtained a grant “Declic Jeunes” from the “Fondation de France” to help in the development of the platform. He is the founder and president of the non-profit organization Science Unchained created in 2020.

Estelle Rascol is an associate professor in medicinal chemistry at the University of Bordeaux since 2018. She is co-founder and secretary of the non-profit organization Science Unchained. She has been supporting GAFFEX from the very beginning since the idea arose during her and Gilmary’s PhD.