Appel à projets doc *iSiM* 2023

Amplification of molecule-induced chirality in CdSe nanoplatelets composite assemblies for magneto-chiral properties.

SPOTLESS

(**s**eeking **p**roperties in nan**o**pla**t**e**l**ets compo**s**ite as**s**emblies)

Summary of the project
The objective of this project is to manipulate optical dichroic properties of CdSe nanoplatelets by triggering their assembly into helicoidal stacks thanks to intercalated chiral molecules and magnetic coordination compounds. Stacked CdSe nanoplatelets are known to propagate an energy input along the direction of stacking. The assemblies we propose will thus lead to composite materials with synergetic magneto-optical properties.

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SCIENCES SORBONNE UNIVERSITÉ   cnrs

Carreer and Education

2018: *Habilitation à diriger des recherches*: *Molecular and Hybrid Materials*
2010-today: Maître de conférences, IPCM, Research group on molecular materials and spectroscopies (ERMMES)
2008-2010: Invited researcher, KIT Karlsruhe, Functional Molecular Nanostructures group of Prof. Mario Ruben
2006-2008: Post-doctoral fellow, CEA Grenoble, Integration of hybrid polyoxometalates into Flash memory devices, under the supervision of Dr. Gérard Bidan
2003-2006: PhD, *Grafting of molecular nanomagnets and coordination networks on Si(100)*, Université Paris-Sud, ICMMO, Orsay, under the direction of Prof. Talal Mallah and Dr. Vincent Huc

Involvements in PhD supervisions: Since 2010, I supervised 3 PhD students: Y. Flores (2015-2018), M. Fuchs (2017-2020), L. Curti (2020-2023). I have been invited to 5 PhD juries outside IPCM (3 times as a “rapporteur” including 2 PhD abroad, and twice in France as an “examinateur”).
Research projects: I have been involved as a partner in 2 Labex projects (2013 & 2016) and the ANR project CocosMen (2018-2023, hold by F. Ribot, LCMCP). I hold 2 projects: Emergence SU (2015) and the ANR project HSP (2016-2021, LOMA in Bordeaux, LCMCP, INSP, ITODYS in Paris as partners).
Foreign collaborations: After a stay at the University of Twente (NL) in 2016, I developed a collaboration with Prof. N. Katsonis and T. Kudernac (today in Groningen) that lead to an excellence grant for Y. Flores and a Hubert Curien project Van Gogh for exchanges of students between the two groups.
Scientific network: Since 2010 I have been a member of the GDR “Magnetism and molecular commutation” and a member of the French association of molecular magnetism since 2020. In that frame, I co-organized the national scientific days of the association in 2022.
Other responsibilities: member of the scientific council of the Chemistry department since 2022 and member of recruitment juries (for assistant professors) in 2019 (SU) and 2023 (U. Paris XIII)

5 significant publications for this project

- *Magnetic Imaging of Cyanide-Bridged Coordination Nanoparticles Grafted on FIB- Patterned Si Substrates. Small,* **2008**, 4, 2240
- *Synthesis, structure and magnetic properties of phenylhydroxamate-based coordination clusters. Dalton Trans*., **2014**, 43, 16805
- *Soft Magnets from the Self-Organization of Magnetic Nanoparticles in Twisted Liquid Crystals. Angew. Chem. Int. Ed.,* **2014***, 53, 12446*
- *Versatile nano-platforms for hybrid systems: expressing spin-transition behavior on nanoparticles. J. Mater. Chem. C,* **2015**, 3, 3350
- *Enhancing the magnetic anisotropy of maghemite nanoparticles via the surface coordination of molecular complexes*. *Nature Communications,* **2015**, 6:10139, DOI: 10.1038/ncomms10139

Laurent COOLEN

Born : 1981, student of ENS Paris : 2000-2004, PhD in Solid States Physics : 2006
Assistant professor (maître de conferences), Sorbonne Université : 2007
Research in nanophotonics and quantum optics, Institut de NanoSciences de Paris (INSP)
Habilitation à Diriger les Recherches (HDR) : 2015

Research topics :
Semiconductor fluorescent nanoparticles
Microscopic study of individual nano-emitters, new methods to probe the transition dipole
Energy transfer and collective effects in self-assembled chains of nanoplatelets
Nano-emitters coupled to photonic or plasmonic nanostructures
Plasmonic application to photovoltaics (perovskite solar cells)

Research production :
47 peer-reviewed articles, 18 since 2018

Z. Ouzit et al., ACS Photonics 10, 421 (2023)	Collective blinking in nanoplatelet chains
J. Liu et al., J. Phys. Chem. A 125, 7572 (2021)	Method for single-emitter dipole analysis
J. Liu et al., ACS Photonics 7, 2825 (2020)	Dipole analysis on nanoplatelet chains
J. Liu et al., Nano Lett. 20, 3465 (2020)	Long range energy transfer in NPL chains
F. Feng et al., ACS Photonics 5, 1994 (2018)	Dipole analysis for single cubic NPLs

Research management :
Co-advisor for 1 thesis (C. Lethiec, 2011-2014, 7 papers)
Advisor for 4 PhD theses (Fu Feng, 2013-2016, 6 papers ; Jiawen Liu, 2017-2020, 7 papers ; Z. Ouzit, 2020-2023, 5 papers ; G. Baillard, 2021-2024)
Co-advisor for 1 post-doc (D. Zheng, 2022-2024)

Research contracts (principal investigator) :
ANR project Foenics (2021-2024, 316 k€ ; with ENS de Lyon)
CNRS PICS international project (2014-2017, France-Vietnam, 18 k€)
ANR project Ponimi (2013-2016, JCJC, 264 k€)
C'Nano project NanoPlasmAA (2009-2012, 83 k€ ; with ESPCI)

Scientific reviewer/expert :
PhD jurys : 8 jurys outside INSP since 2016, 5 as rapporteur
Member of CNU (section 30, 2012-2019), board member (2015-2019)
Co-chairman DPC 16, abstract selection + guest editor J. Lumin (2016)
Reviewer for ANR (2011, 2021, 2023), Labew PALM Paris-Saclay (2022), NCN (Poland, 2016)
Recruitment committees : Maître de conferences (Paris 7 (2011), PhD grants (ED PIF, 2018)

Scientific description

Objectives and description of the project

The objective of this project is to manipulate the optical dichroic properties of CdSe nanoplatelets by triggering their assembly into helicoidal stacks thanks to intercalated chiral molecules and magnetic coordination compounds. Stacked CdSe nanoplatelets are known to propagate an energy input along the direction of stacking.[1] The assemblies we propose will thus lead to composite materials with synergetic magneto-optical properties.
CdSe nanoplatelets are semiconducting nanocrystals. The thickness of the platelets can be controlled at the atomic layer scale (from 2 to 8 layers of Se atoms) during their synthesis.[2–8] That results in 1D quantum confinement that provides CdSe nanoplatelets unique optical properties such as unparalleled spectrally sharp and monodisperse luminescence in the visible region at discrete wavelength corresponding to their thickness. These nanoparticles will be functionalised by chiral molecules that will play a triple role: (*i*) transferring chirality to the nanoparticles for dichroic optical properties, (*ii*) triggering the assembly of the nanoplatelets into helicoidal stacks to amplify the properties, (*iii*) bring paramagnetism for magneto-chiral properties.

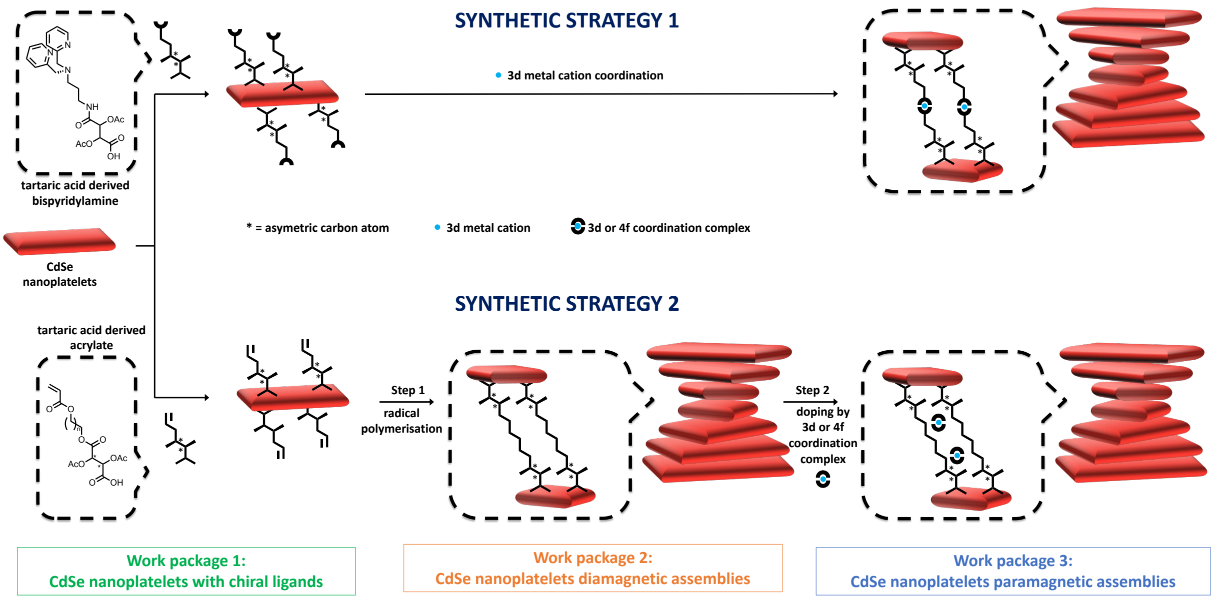

Figure 1: schematic over-view of the project

Two synthetic strategies will be followed in order to fulfil five work packages (figure 1). The starting common point in both strategies is the coordination of tartrate derivatives at the surface of CdSe nanoplatelets.

Synthetic strategy 1: assembly by coordination polymerisation

The tartrate ligands will be tailored so as to bear a second free coordination site able to link the nanoplatelets face-to-face thanks to metalation with paramagnetic transition metal ions.

Synthetic strategy 2: assembly by organic polymerisation

Step 1: The tartrate ligands will be tailored so as to bear an acrylate function in order to link the nanoplatelets face-to-face by radical polymerisation.
Step 2: Paramagnetic complexes will diffuse into the super-structure to complete the composite material.

Work Package 1 (WP1): CdSe nanoplatelets with chiral ligands (IPCM & INSP)

Task 1.1: Tartrate derivatives will be prepared in one step from commercially available (R,R) and (S,S) diacetyl tartaric anhydrides: (*i*) by amidification with bis(pyridin-2-ylmethyl)propane-

1,3-diamine (strategy 1), and (*ii*) by esterification of commercially available 4-hydroxybutyl acrylate and 2-hydroxyethyl acrylate (strategy 2). All ligands will be prepared and characterised (^{1}H and ^{13}C NMR, mass spectroscopy) at IPCM.

Task 1.2: Reacting Cd myristate, selenium and oleic acid in octadecene yields CdSe nanoplatelets whose thickness depends on the reaction conditions. After purification, the nanoplatelets will be covered with oleate ligands and dispersed in non-polar solvents such as toluene or hexane.

Task 1.3: The oleate ligands covering the CdSe nanoparticles can easily be exchanged by other ligands in solution.[9] Reacting an excess of molecules prepared in WP1 displaces the ligand exchange equilibrium towards the target hybrid nanoplatelets.
CdSe nanoplatelets will be synthesised and characterised by UV-visible and photoluminescence spectroscopies at IPCM and TEM on the microscopy platform of the Chemistry department. These techniques also allow the monitoring of the ligand exchange reaction.

Task 1.4: The luminescence properties of the nanoplatelets will be analyzed at INSP, either in solution or at the scale of individual emitters by fluorescence microscopy. Their quality will be inferred from their quantum yield, emission intermittency (blinking) and dynamics (decay curves). The effect of the ligands will be analyzed on the emission circular polarization (CPL).

Work Package 2 (WP2): Diamagnetic assemblies (IPCM & INSP)

Task 2.1: Following synthetic strategy 2, acrylate-terminated nanoparticles will be polymerised under UV irradiation (365 nm) using a photoinitiator. This polymerisation will lead to helicoidal superstructures made of stacks of chiral nanoplatelets (IPCM). The morphology of the stacks will be checked by TEM. Small angle X-ray scattering (SAXS) can also be performed at ENS Lyon in the group of B. Abécassis for the measurement of stacks length and inter-particle distances.

Task 2.2: At INSP, the luminescence of the platelet assemblies will be compared to the luminescence of the isolated platelets. The quality of the assemblies and platelet protection by the ligands will be probed through quantum yield or decay dynamics. The efficiency of energy transfer between platelets will be probed, as in ref. 1, by imaging the luminescence from a platelet chain excited in one spot and measuring the extension of the emission portion. The molecular assembly will offer the possibility to vary the distance between platelets via the length of the molecules. A strong effect ($1/d^2$) of this distance is expected on the length of the energy transfer diffusion over the chain, so that propagation lengths up to 1 µm or more may be obtained.

Work Package 3 (WP3): Magnetic assemblies (IPCM & INSP)

Task 3.1: Following synthetic strategy 1, bis(pyridinylmethyl)amine-covered nanoplatelets will be reacted with different 3d metal ions to trigger their assembly by coordination chemistry. This reaction will lead to helicoidal superstructures made of stacks of chiral nanoplatelets with intercalated magnetic coordination complexes.

Task 3.2: The last step of synthetic strategy 2 consists in diffusing magnetic (3d or 4f) coordination complexes into the superstructures obtained in WP2 in order to get lamellar and magnetic helicoidal stacks of chiral nanoplatelets.

The same chemical characterisation methods as in work packages 1&2 will be employed. An in-depth study of the physical properties of the target assemblies will also be performed in order (*i*) to elucidate the consequences of combined chirality and magnetism on their optical

properties and (*ii*) to detect a possible enhancement of these properties in comparison with the isolated nanoparticles.
Task 3.3: The emission polarization of the assemblies will be analyzed at the scale of a single chain, deposited on a glass substrate, and the circular polarization will be probed.

Task 3.4: Magnetic circular dichroism (MCD) on solutions of stacked particles can be performed at IPCM with the help of INSP by adapting a magnet on our CD spectrometer.[10] If needed, IPCM recently acquired a cryostat to be adapted to various spectrometers for low-temperature measurements. In addition, element-specific contribution to these properties can also be measured by X-ray magnetic circular dichroism (XMCD) on the Deimos beam line at Soleil synchrotron since the ERMMES group of IPCM is expert in this technique.

Originality of the project

- Functionalisation of CdSe nanoplatelets with chiral ligands has been described with various thiolated molecules but leads to the loss of their luminescence and their CD spectra reveals moderate dissymmetry factors (*g*-factors ≈ 10^{-4}).[11–15] On the contrary, we recently demonstrated that the functionalisation of CdSe nanoplatelets with simple aliphatic tartrate derivatives (PhD thesis of L. Curti, to be defended) keeps the luminescence of the particles and leads to exceptional *g*-factors (2.10^{-3}).
- Achieving long and stable stacks of CdSe nanoplatelets covered by native oleate ligands is difficult and somehow difficult to reproduce because the assembly is driven by weak interactions.[16–19] The use of coordination links or covalent links to secure the assemblies has never been described and will help manipulating such superstructures for integration in future devices.
- Control of the chirality in assemblies of CdSe nanoplatelets has never been described since no source of chirality has never been introduced in such systems. The assembly of CdSe nanoplatelets in helicoidal stacks has been described by playing on the mechanical strain induced by the ligands on the nanoparticles, but this strategy leads to racemic mixtures.[20] On the contrary, our approach is based on chiral molecules to induce such a stereoselectivity on the assemblies.
- Synergy between magnetism, chirality and optical properties belongs to molecular chemistry.[21] In our approach we expect enhanced properties thanks to the assembly process with regards to the properties of isolated hybrid nanoplatelets. Our approach towards hybrid and composite materials is thus disruptive since it can produce applicative nano-objects.
- Studying interactions between nanoparticles is often complex as it requires a good knowledge of the assembly of the particles. Self-assembled chains of platelets are a unique model system to analyze such mechanisms. The analysis of Förster transfer (FRET) in such systems, as pioneered in ref. 1, will take a new turn with the level of control offered by molecular methods of assembly.

Feasibility. Risk assessment and management

From a general point of view, this project is new and ambitious. We are nonetheless confident in its success since synthetic difficulties have already been evaluated during L. Curti PhD (2019-2023) and a master internship in 2022. In addition, offering 2 alternative strategies for the chiral chain assembly mitigates the risk.

WP1 does not present any risk. Organic Chemistry implies one step reactions on the gram scale and without any tedious purifications. For instance, the esterification of tartaric anhydride has been achieved during the PhD of L. Curti at IPCM with several aliphatic alcohols. The same way, the synthesis and use of CdSe nanoplatelets has been studied at IPCM during the PhD of. L. Curti.

WP2 is innovative and presents more risk. In particular, we will dedicate time to optimise the experimental conditions during polymerisation, in particular concentration, in order to avoid reticulation that would lead to branched assemblies. In the worst case, bulk polymers or gels of nanoplatelets would be obtained. That case, even if not desired, would give nevertheless interesting materials bearing the same properties as expected for the target super structures and would thus lead to original publications.

Regarding **WP3**, the risk of **task 3.2** is the possible difficulty of "doping" the super structures obtained after WP2 with coordination complexes. The choice of the complex (size, charge) will be studied as well as the synthetic conditions (concentration, temperature) in order to maximize the number of complexes to intercalate in the structure. The advantage is the use of the polymeric chiral matrix of CdSe nanoplatelets obtained in WP2. Indeed, such a matrix should be more stable than van der Waals-stabilised assemblies of nanoplatelets in a wider range of synthetic conditions.

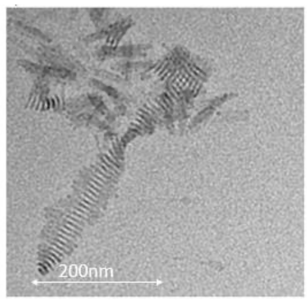

Figure 2: helical assembly of CdSe nanoplatelets with chiral ligands and CoPc complexes. The stack is 237nm long with a 3 nm inter-particle distance.

The risk of **task 3.1** has been evaluated during a master internship in 2022. This preliminary work involved chiral tartrate derivatives on CdSe nanoplatelets terminated by nitrile ligands able to coordinate apical position of Co(II) phthalocyanines (CoPc). Helical assemblies of CdSe nanoplatelets were detected by TEM (figure 2) but the assemblies are short, probably because of the lability of the apical positions of the Co(II) ion, coordinated to weak nitrile ligands. This is why we chose a bispyridinyl amino ligand to be reacted with a 3d metal salt for a stronger chelate effect to stabilise the assembly. In addition, one could fear the quenching of CdSe luminescence by the intercalated magnetic 3d ions. In the case of CoPc mentioned above, the presence of the Co(II) complex was confirmed by UV-visible spectroscopy and the luminescence was nevertheless maintained. This is why we will use Co(II) complexes at first for this WP. **Tasks 3.3 and 3.4** are not risky since IPCM and INSP are equipped with adequate spectrometers and microscopes. It will nevertheless be time consuming to optimise sample preparation and results interpretations.

Position of the project within national and international context

CdSe nanoplatelets have been described for the first time in 2008.[22] This project is therefore at the core of a brand new and hot domain with several international groups intensively producing publications. Some of these groups are French and IPCM and INSP both benefit from an ongoing collaboration at ENS Lyon with the group of B. Abécassis who is expert in nanoplatelets synthesis, ligand exchanges, self-assembly and physical characterisations. This active collaboration is an asset for the success of our project and will be enriched during the coming years. More locally, intense exchanges were recently born between INSP groups and B. Fleury at IPCM, revealing the need of both communities to enrich each other (an ANR project has just been accepted in phase 1 between B. Gallas at INSP and B. Fleury for the design of a nano and ultra-sensitive CD spectrometer. Chiral CdSe nanoplatelets produced at IPCM will be used as reference samples for this spectrometer. Platelet assembly, as described here, is outside the scope of this ANR proposal). Indeed, the optical properties of CdSe nanoparticles strongly depend on their surface. Molecular chemists can thus bring a strong input in the domain by designing and synthesising new ligands and assembly protocols for these nanoparticles. Therefore, we strongly believe that the success of this project would be extremely federative for the French groups working on the physico-chemistry of 2D nanomaterials.

Position of the project in regard to the iSiM objectives

This project is molecule-centred. It strongly depends on chiral organic ligands and magnetic coordination complexes. In this project, the molecules aim at structuring nanoplatelets into supra-particular nano-objects. The molecules will also bring properties since we seek a synergy between optical properties (absorption and luminescence) of CdSe nanoplatelets on the one side and chirality and magnetism of molecules on the other side. The induction of chirality in semiconducting nanocrystals, combined to their outstanding optical properties, represents a way to access a new manifold of possible applications, such as stereoselective synthesis,[23–25] chiral recognition,[26] biosensing,[27–30] asymmetric catalysis and display devices.[31,32] In addition, it could also address the question of the cosmic origins of the molecular bricks of living organisms. Indeed, a strong synergy between chirality, optical and magnetic properties can result in magneto-chiral dichroism, that is dichroic properties of chiral magnetic compounds excited by non-polarised light and the control of this dichroism by the direction of an applied magnetic field.[21,33,34] This property has been proposed as the cause of homochirality of of amino-acids on Earth.[35,36] The quest of this rare property lies beyond the scope of this project and should be explored on our compounds thanks to a wider consortium. The success of this project would thus be an asset for a subsequent proposal in another call for proposal at the national level (ANR).

Highlight partners complementarity

The goup of L. Coolen developed various methods to analyze the radiation of individual luminescent nano-emitters by fluorescence microscopy and showed that CdSe/CdS nanoplatelets present an asymmetric emission in terms of polarisation.[37,38] His recent work dealt with linear assemblies of nanoplatelets achieved at ENS Lyon.[1] He showed that these systems present a very fast energy transfer (a few ps) leading to the energy diffusion along a hundred of platelets. This energy transfer plays a crucial role on the luminescence properties of the assemblies: quantum yield, multi-excitonic cascades, laser effect, etc. The understanding of this energy transfer would guide the development of new opto-electronic systems. Nanoplatelets assemblies bearing a controlled chirality have never been reported in the literature and open new perspectives in this domain.
The research activities of Benoit Fleury are focused on the study of confined molecular systems at interfaces and polyfunctional molecular and hybrid systems. He belongs to the ERMMES group (molecular materials and spectroscopies), which has an international visibility on coordination chemistry. B. Fleury's work in this group revolves around two intersecting axes: the integration of magnetic molecules into composite materials on the one hand and, on the other hand, the exaltation of molecular properties to the macroscopic scale.
As a result, for this project dealing with the synthesis of hybrid and composite materials with optical properties, the partners have chosen to associate the competencies in Physics of L. Coolen who is expert in the measurement of CdSe nanocrystals properties to the competencies in Chemistry of B. Fleury who is specialized in the synthesis of hybrid nanoparticles.

Bibliography

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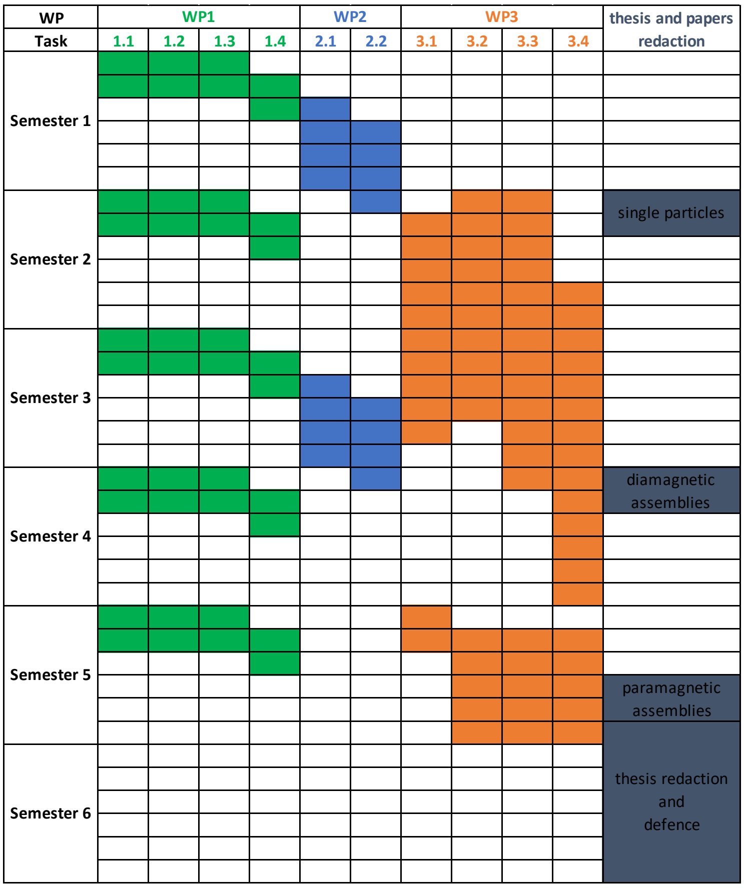
Research program



This Gantt diagram presents an over-view of the scheduled research program.

WP1 being riskless, all synthesis will be performed on-demand all along the project duration.

WP2 will require a focus attention on longer time scales in order to optimize chemical reactions and physical measurement conditions.

WP3 will be time demanding in order to optimize chemical reactions, physical measurement conditions and cross-checking results between different physical methods.

Enough time will also be dedicated to papers redaction to disseminate results about individual particles and their assemblies. The last semester will mainly be dedicated to the PhD thesis redaction and defence preparation.