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Non photochemical light induced nucléation

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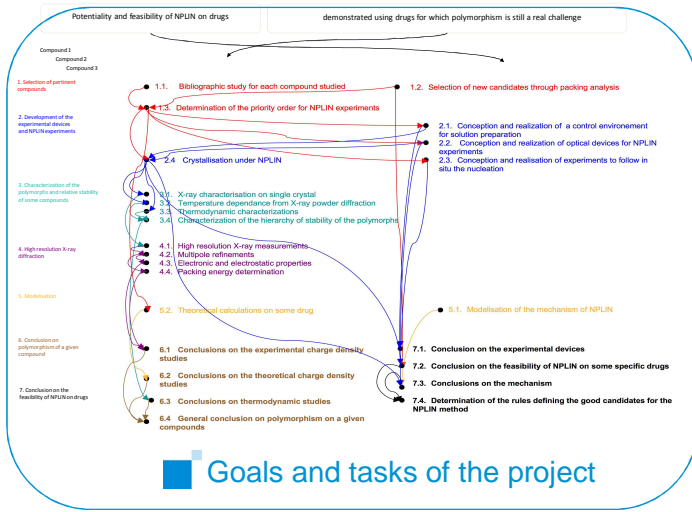
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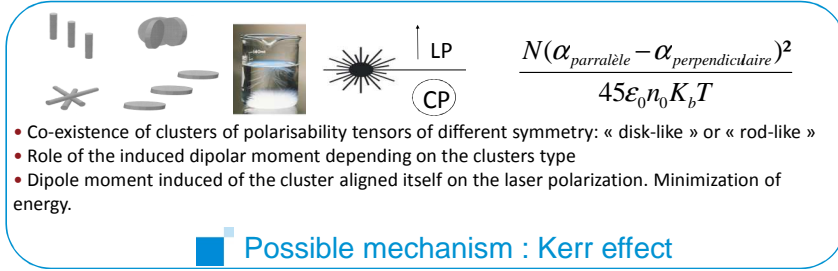


Polymorphism studies important in the industrial pharmaceutical context

Method developed on urea [1], glycine [2], L-histidine [3], Hen Egg White Lysozyme [4], Bovine P T [5], Pentyl cyanobiphenyl [6], KCl [7] [9], NaClO₃ [8], KBr [9], Acetic acid [10]

[1] Garett B.A. et al. *Phys. Rev. Lett.* **1996**, 16, 3475-3476; Yoshikawa H.Y. et al. *Jpn J. Appl. Phys.* **2006**, 45, 302-305; Yoshikawa H.Y. et al. *Jpn J. Appl. Phys.* **2006**, L23-L26. [2] Zaccaro J., et al. *Cryst. Growth & Des.* **2001**, 1, 5-8; Garett B.A. et al. *Phys. Rev. Lett.* **2002**, 17, Matic J. Ph.D. **2005**; Matic J. et al. *Cryst. Growth Des.* **2005**, 5, 1565-1567; Sun X.Y. et al. *Cryst. Growth Des.* **2005**, 5, 684-689; Sugiyama T. et al. *Chem. Lett.* **2007**, 12, 1489-1491; Sun X.Y. Ph.D. **2008**; Sugiyama T. et al. *Chem. Lett.* **2009**, 15, 482-483. [3] Sun X.Y. et al. *Cryst. Growth Des.* **2008**, 8, 1720-1722. [4] Lee et al. *Crystal Growth & Design* vol.8 **2008** n°12, 4255-4261 [5] Lee et al. *Crystal Growth & Design* vol.8 **2008** n°12, 4255-4261 [6] Sun et al., *Phys. Rev. E* vol.79 **2009** 021701 [7] Alexander A.J. et al. *Cryst. Growth Des.* **2009**, 9, 958-963; Duffus A.J. et al. *J. Am. Chem. Soc.* **2009**, 131, 11676-11677; Ward M.R. et al. *Phys. Rev. Lett.* **2009**, 481, 25-28. [8] Ward et al. *J. Chem. Phys.* Vol. **135** **2011** 114508 [9] Ward et al. *Crystal Growth & Design* vol.12 **2012**, 4554-4561 [10] Ward et al. *Phys. Chem. Chem. Phys.* **2011** Vol.14

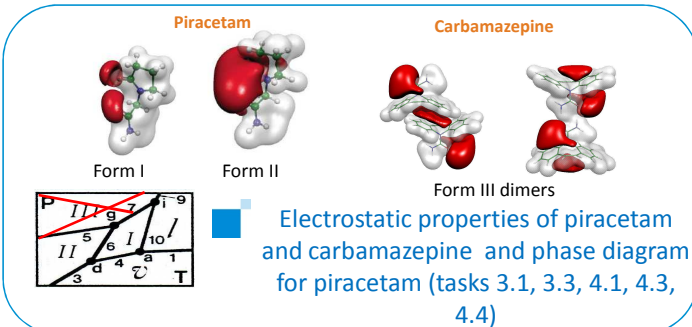
Context



$$N(\alpha_{\text{parallèle}} - \alpha_{\text{perpendiculaire}})^2 = 45\epsilon_0 n_0 K_b T$$

- Co-existence of clusters of polarisability tensors of different symmetry: « disk-like » or « rod-like »
- Role of the induced dipolar moment depending on the clusters type
- Dipole moment induced of the cluster aligned itself on the laser polarization. Minimization of energy.

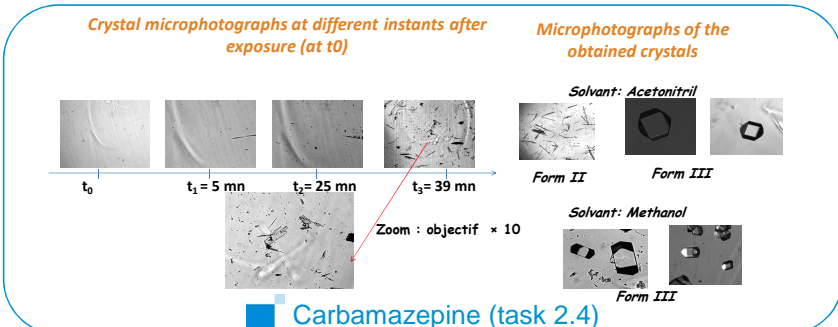
Possible mechanism : Kerr effect



Piracetam Form I, Form II

Carbamazepine Form III dimers

Electrostatic properties of piracetam and carbamazepine and phase diagram for piracetam (tasks 3.1, 3.3, 4.1, 4.3, 4.4)



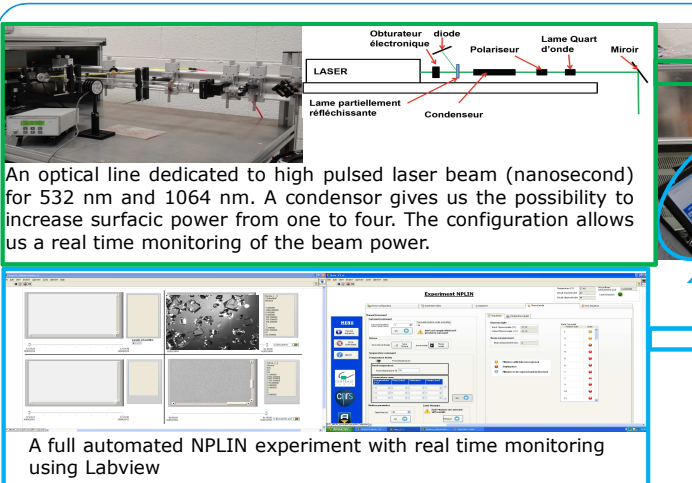
Crystal microphotographs at different instants after exposure (at t0)

Microphotographs of the obtained crystals

Solvent: Acetonitril (Form II, Form III)

Solvent: Methanol (Form III)

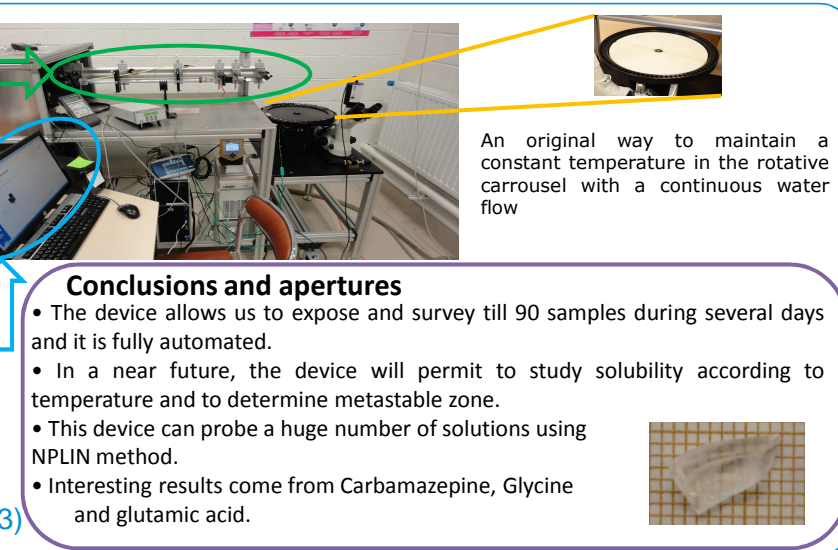
Carbamazepine (task 2.4)



NPLIN new experimental device (task 2.1, 2.2, 2.3)

An optical line dedicated to high pulsed laser beam (nanosecond) for 532 nm and 1064 nm. A condensor gives us the possibility to increase surfacic power from one to four. The configuration allows us a real time monitoring of the beam power.

A full automated NPLIN experiment with real time monitoring using Labview



Conclusions and apertures

- The device allows us to expose and survey till 90 samples during several days and it is fully automated.
- In a near future, the device will permit to study solubility according to temperature and to determine metastable zone.
- This device can probe a huge number of solutions using NPLIN method.
- Interesting results come from Carbamazepine, Glycine and glutamic acid.

Production scientifique (publications, brevets)

- B. CLAIR, A. IKNI, H. YANG, F. DUMAS, P. SCOUFFLAIRE, A. SPASOJEVIC – DE BIRÉ « Non Photochemical Light Induced Nucleation. A tool to crystallize polymorphs on demand ? » CPOSS meeting, London (UK), Grande-Bretagne, 30 mars 2011
- A. IKNI, B. CLAIR, H. YANG, F. DUMAS, P. SCOUFFLAIRE, A. SPASOJEVIC – DE BIRÉ « Non Photochemical Light Induced Nucleation. A tool to crystallize polymorphs on demand ? » IV congrès algérien de cristallographie, Kenchela, (Algérie), 14-16 mai 2011
- A. SPASOJEVIC – DE BIRÉ, A. IKNI, B. CLAIR, N. GUILBIN, I. KORNEV, X. SHI, J. LONG, H. YANG, W. LI, M. MENDRE, P. SCOUFFLAIRE, E. JEAN-BART, J. BEAUNIER, F. SAIG, P. ESPEAU, Y. CORVIS, C. ALZINA, N-E GHERMANI, N. EL HASSAN, F. DUMAS, S. VEESLER, Z. HAMMADI « Non Photochemical Light Induced Nucleation » Journées nationales en nanosciences et nanotechnologies, Strasbourg (France) 7 – 9 novembre 2011
- A. SPASOJEVIC – DE BIRÉ, A. IKNI, B. CLAIR, F. DUMAS, P. SCOUFFLAIRE, « Non Photochemical Light Induced Nucleation (NPLIN). A tool to crystallize polymorphs on demand ? Application to carbamazepine » GFCC-2011, Paris, novembre 2011
- B. CLAIR, A. IKNI, P. SCOUFFLAIRE, S. VEESLER, A. SPASOJEVIC – DE BIRÉ « A Method to probe NPLIN on glycine and histidine : a new path to understand nucleation » CPOSS meeting, London (UK), 3 avril 2012
- A. IKNI, B. CLAIR, P. SCOUFFLAIRE, S. VEESLER, A. SPASOJEVIC – DE BIRÉ, « Non Photochemical Light Induced Nucleation (NPLIN). Application to carbamazepine (CBZ) » CPOSS meeting, London (UK), 3 avril 2012
- A. SPASOJEVIC – DE BIRÉ, P. SCOUFFLAIRE, A. IKNI, B. CLAIR, E. JEAN-BART, J. BEAUNIER, F. SAIG « New automated experimental devices for Non Photochemical Light Induced Nucleation » CPOSS meeting, London (UK), 3 avril 2012
- B. CLAIR, A. IKNI, P. SCOUFFLAIRE, A. SPASOJEVIC – DE BIRÉ, « NPLIN of glycine and carbamazepine » Serbian Crystallographic Meeting, Bela Crkva (Serbie), 31 mai – 2 juin 2012
- A. IKNI, B. CLAIR, P. SCOUFFLAIRE, A. SPASOJEVIC – DE BIRÉ, « NonPhotochemical Light Induced Nucleation of Carbamazepine in Polar protic and aprotic solvents » 2012 Collaborative Conference on Crystal Growth, Orlando (USA), 11-14 décembre 2012
- B. CLAIR, A. IKNI, P. SCOUFFLAIRE, A. SPASOJEVIC – DE BIRÉ, « Understanding photophysical effects : NPLIN on glycine and L- glutamic acid » 2012 Collaborative Conference on Crystal Growth, Orlando (USA), 11-14 décembre 2012

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