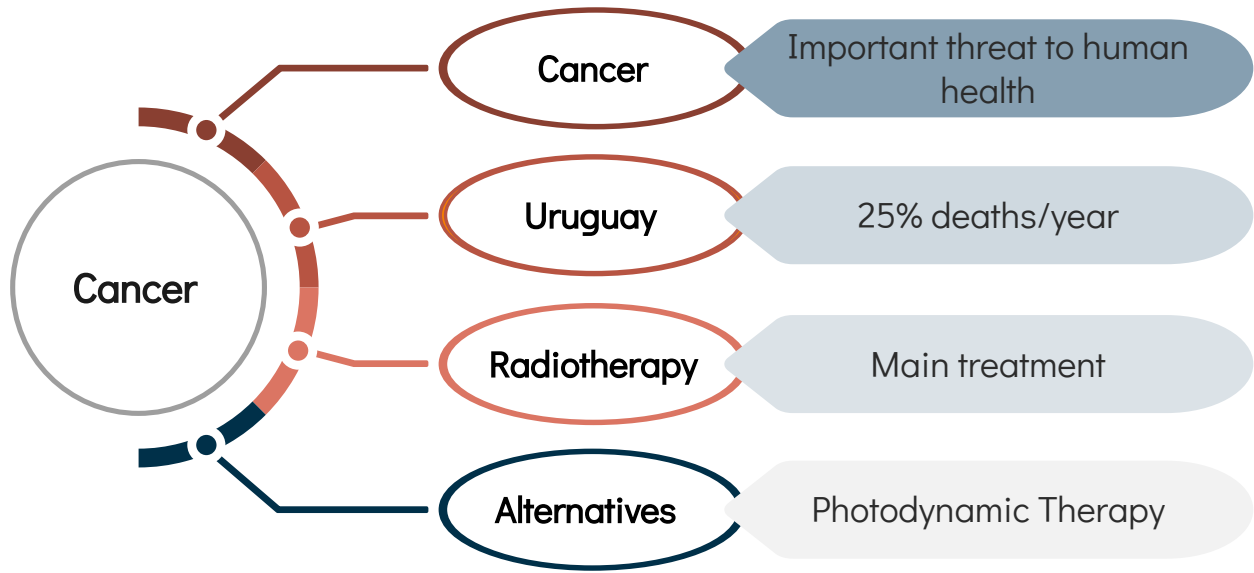


# Fluoro-perovskite nanomaterials for photodynamic cancer treatment

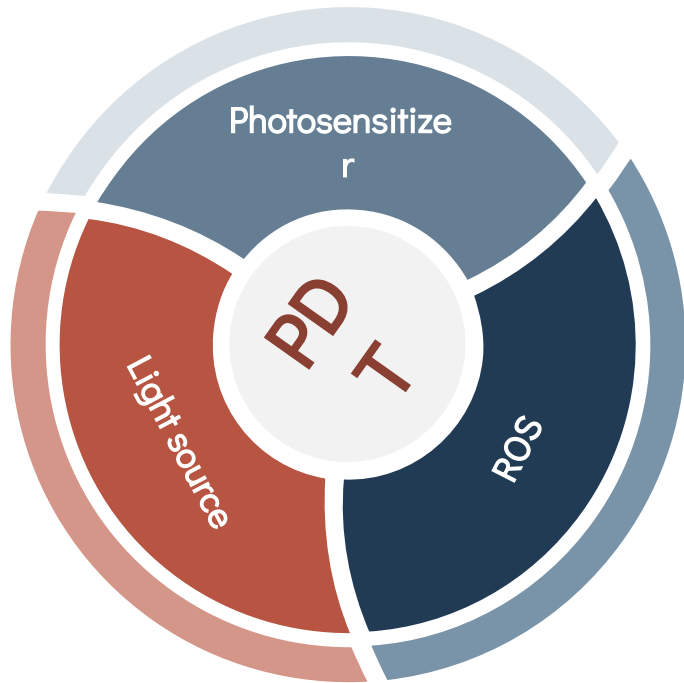
María Eugenia Pérez Barthaburu  
Departamento de Desarrollo Tecnológico  
CURE  
Universidad de la República

Romina Keuchkerian, Leopoldo Suescun, Carolina Crisci, Ivana Aguiar, Wilner  
Martínez López, Mauricio Rodríguez Chialanza

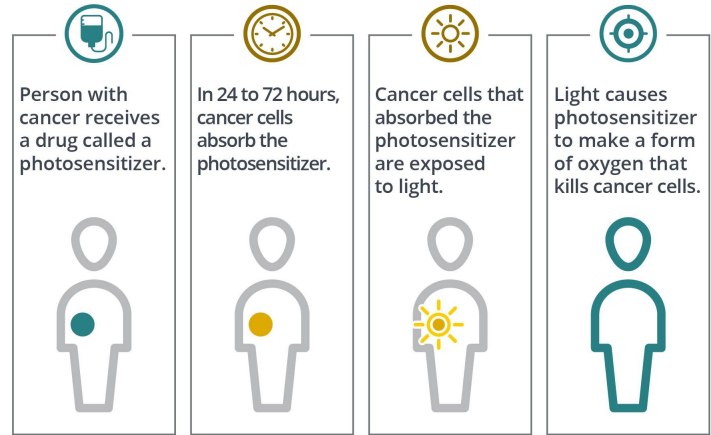
# Background



# Photodynamic Therapy (PDT)

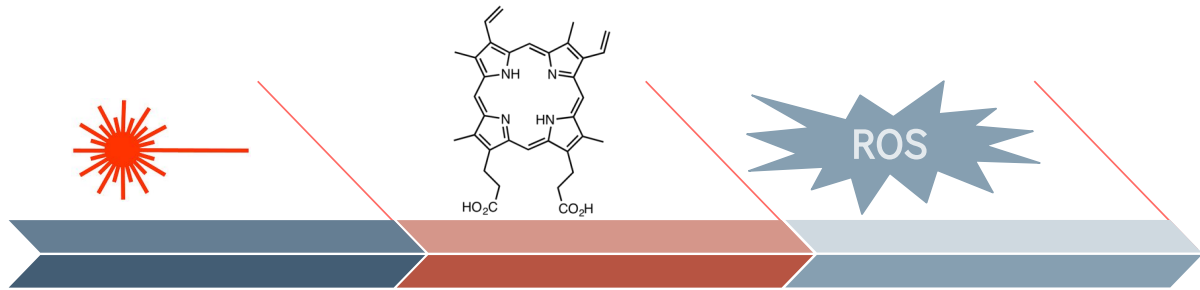


## PHOTODYNAMIC THERAPY



[cancer.gov/about-cancer/treatment/types/photodynamic-therapy](https://www.cancer.gov/about-cancer/treatment/types/photodynamic-therapy)

# Photodynamic Therapy (PDT)



## Laser

Ideally in the  
biological window  
(700-2500 nm, )

## Photosensitizer

UV-Vis activation  
  
Limit the depth of  
tumor

## ROS generation

Generated *in situ*

# Photodynamic Therapy (PDT)

## Limitations

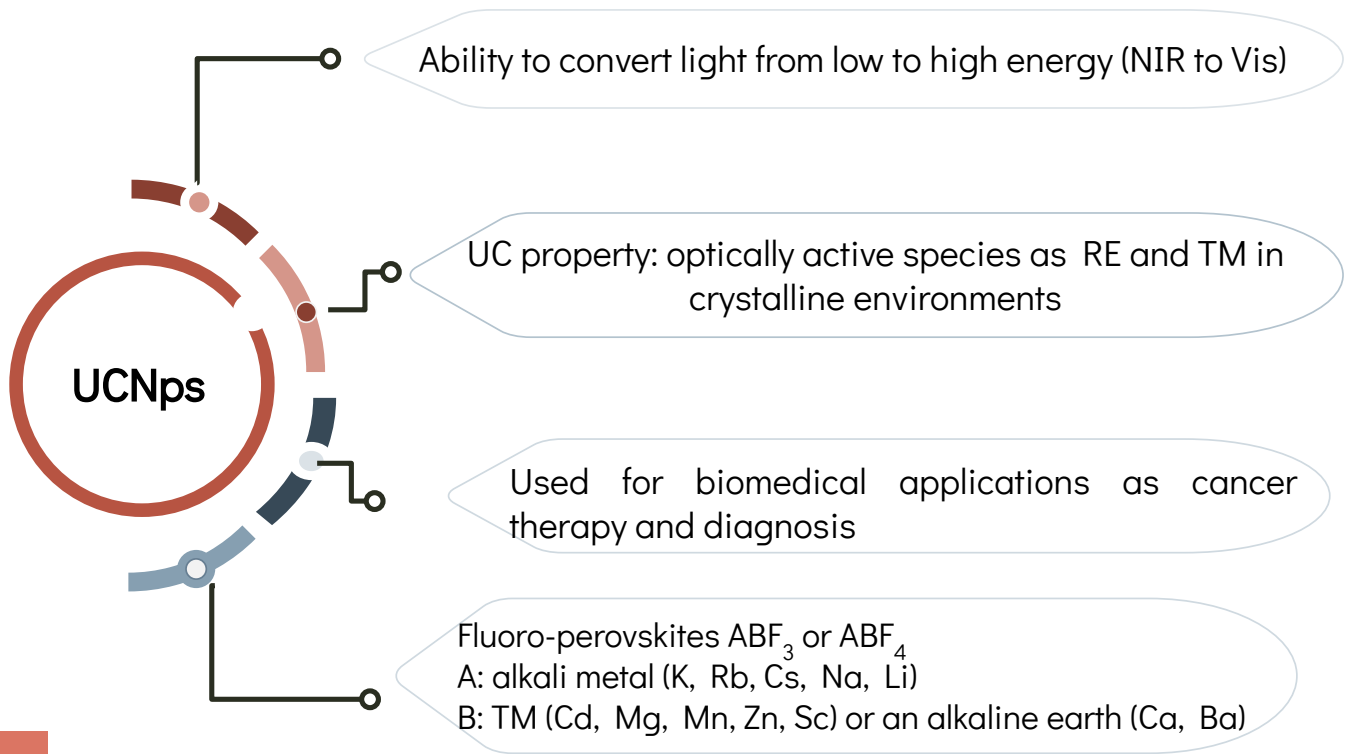
Not in the biological window

Conventional PS are activated with UV-Vis light

Treatment of superficial tumors

Need move invasive procedures to reach throat tumors

# How to extend PDT to deeper tumors



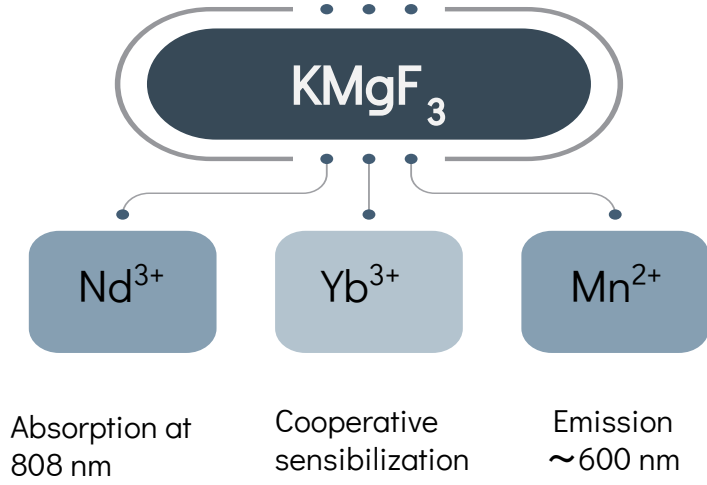
# Our aim



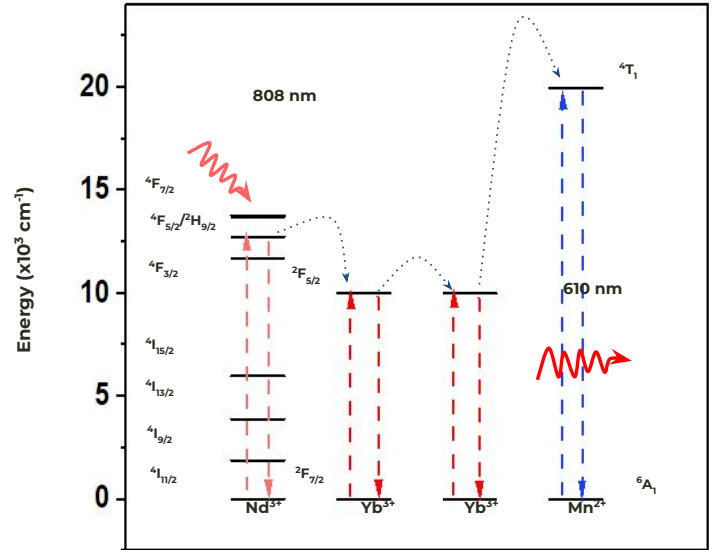
To develop fluoro-perovskites nanoparticles with upconversion properties by the addition of TM and RE as a way of extending PDT to deeper tumors while reducing the use of RE

- *Master in Chemistry Thesis “Desarrollo de nanopartículas para aplicación en terapia fotodinámica”, Romina Keuchkerian*
- *R+D Project “Desarrollo de nanopartículas con propiedad de conversión ascendente para potenciales aplicaciones Biomédicas” FCE-1-2020-162287, resp. Romina Keuchkerian*

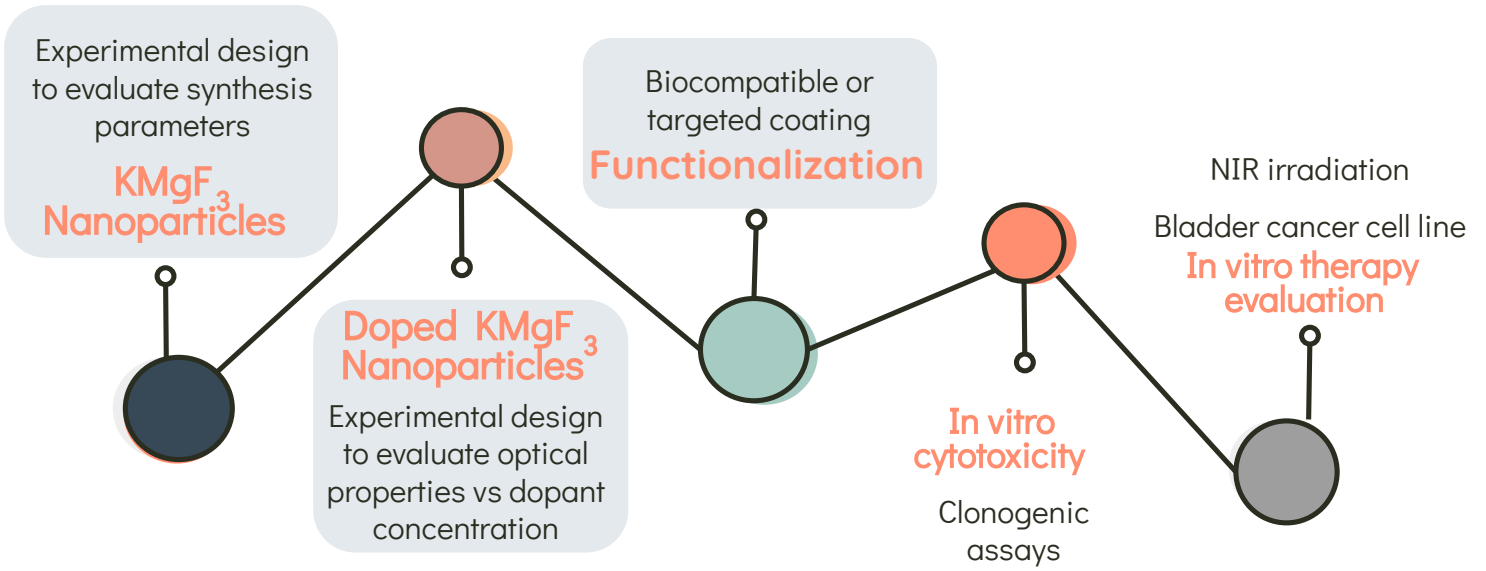
# UCNps

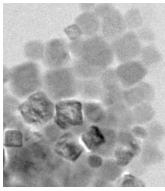


# Our proposal





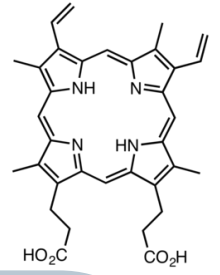




size < 200 nm  
Ideal < 50 nm

UCNps

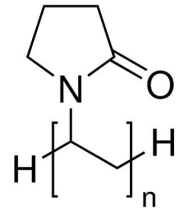
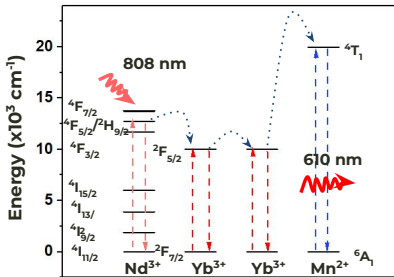
ALA: PpIX 630 nm



KMgF<sub>3</sub>

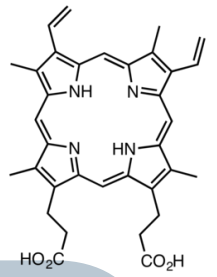
Nd: Yb: Mn

PVP



TEM  
XRD - Scherrer

Experiments in  
vitro  
Cytotoxicity



size < 200 nm  
Ideal < 50 nm

UCNps

ALA:PpIX 630 nm

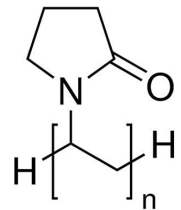
$KMgF_3$

NdYb:Mn

PVP

Optical properties:  
Absorbance  
Photoluminescence

Confirmation  
FTIR



# DoE of the synthesis

DoE 2<sup>4</sup>

## Solvothermal



Reagents:  
 $MgCl_2 \cdot 6H_2O$   
 $NH_4F$   
KOH

Solvent:  
 $H_2O$   
EtOH  
OA

4 factors 2 levels  
16 experiments

Predictor variables

Response variable

Temperature  
Time  
 $[MgCl_2]$   
 $[NH_4F]$

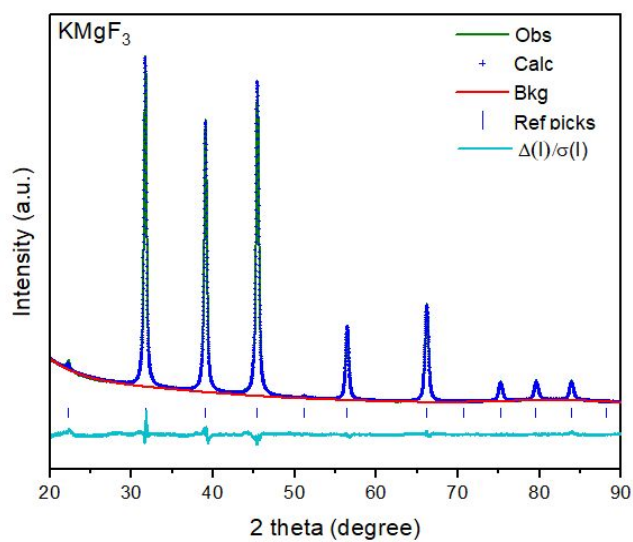
Nps size (XRD)

Multiple linear regression

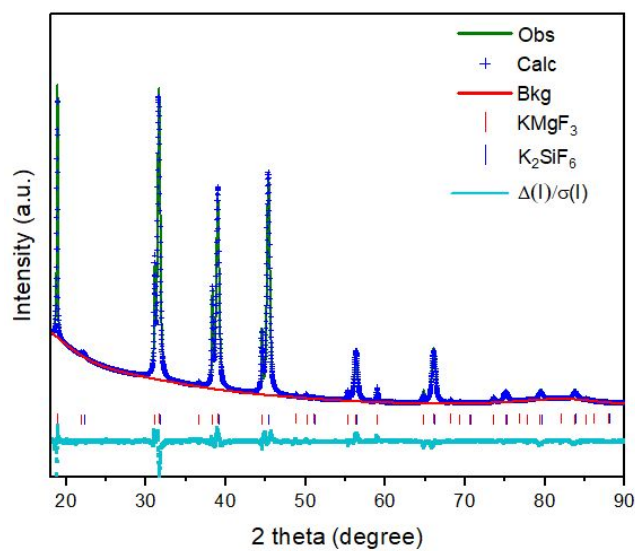


# XRD results

KMgF<sub>3</sub> (Pm-3m) cubic phase

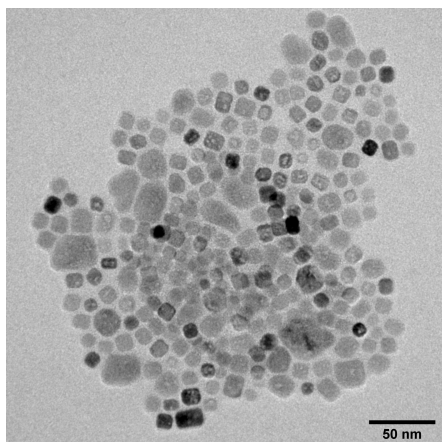


160°C, 24hs, NH<sub>4</sub>F : MgCl<sub>2</sub> / 1:1

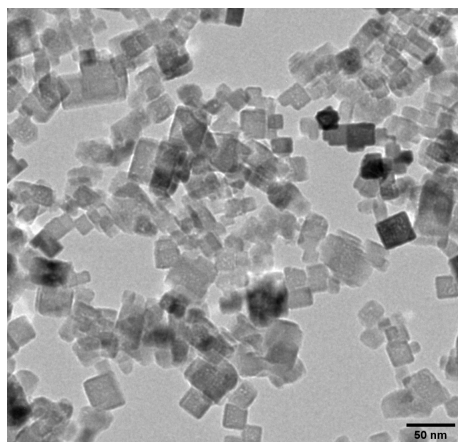


200°C, 6hs, NH<sub>4</sub>F : MgCl<sub>2</sub> / 2:1  
NH<sub>4</sub>F excess

# TEM results



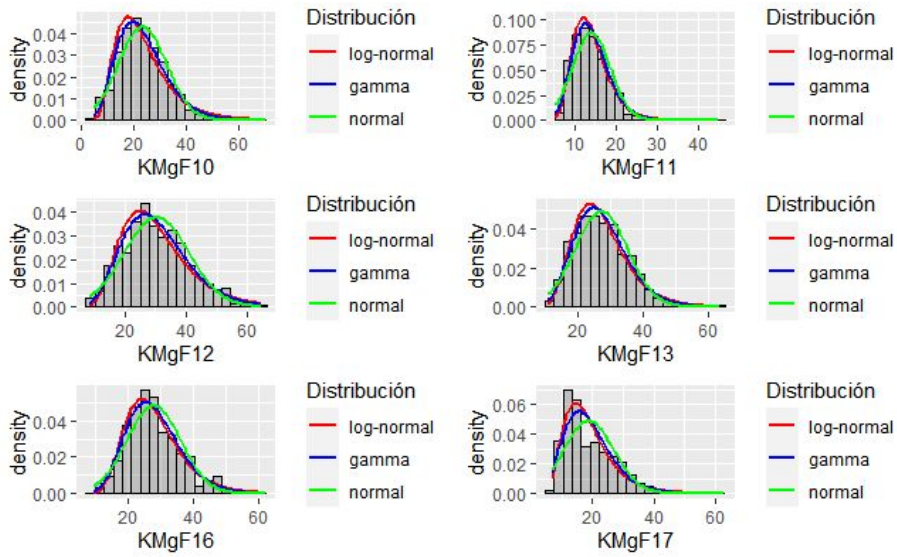
160°C, 6hs,  $\text{NH}_4\text{F} : \text{MgCl}_2 / 1:1$



200°C, 6hs,  $\text{NH}_4\text{F} : \text{MgCl}_2 / 2:1$   
 $\text{NH}_4\text{F}$  excess

- ✓ Square morphology
- ✓ Unit cell size slightly higher for sample with  $\text{NH}_4$  excess

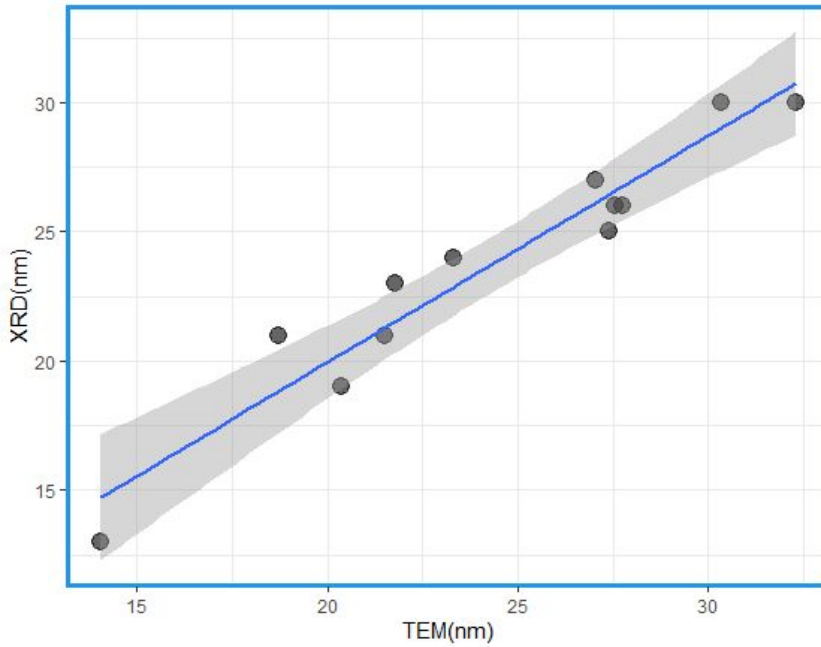
# Size distribution



✓ Evaluated: normal, lognormal and gamma with 0.05 significance level

*Nanotechnology 10 (1999) 25–28*  
*J Nanopart Res (2009) 11:1713–1718*

# Correlation XRD-TEM



- ✓ Pearson correlation coefficient
- ✓ Correlation: 0.9634751
- ✓ p-value:  $4.815E^{-7}$

XRD

$13,46 \pm 0,30$  nm to  $32,18 \pm 0,60$  nm

TEM

$14,22 \pm 4,47$  nm to  $32,38 \pm 9,99$  nm



Further details on statistical analysis visit poster: 4FFR 28/09 18:00h



# Statistics of experiments

## Nanopartículas con conversión ascendente

Coefficients				
	Estimated	Standard error	t value	Pr(> t )
(Intercept)	23.4954	2.0114	11.681	7.78e-10
Temperature	5.7585	2.2191	2.595	0.0183
Time	-3.0797	2.2312	-1.380	0.1844
NH <sub>4</sub> F	-0.3383	2.4922	-0.136	0.8935
MgCl <sub>2</sub>	0.2814	2.0047	0.140	0.8899
Time:NH <sub>4</sub> F	5.7622	3.2003	1.800	0.0886
Temperature:MgCl <sub>2</sub>	-7.3669	3.3078	-2.227	0.0389

- Significant effect of temperature and of interaction between [MgCl<sub>2</sub>] and temperature

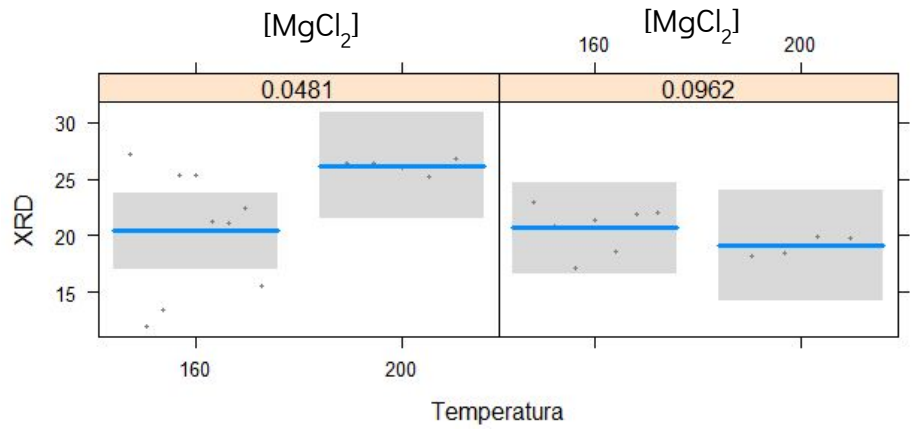
- Significance level: 0.05

# Statistics of experiments

F=0.1522436

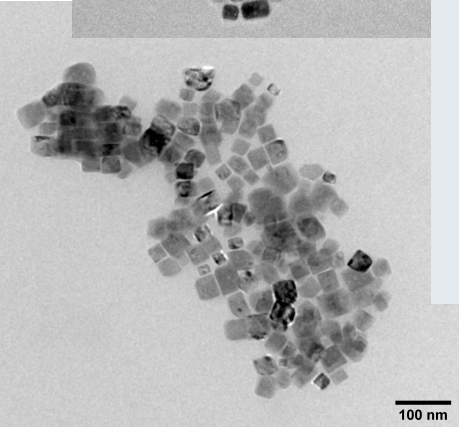
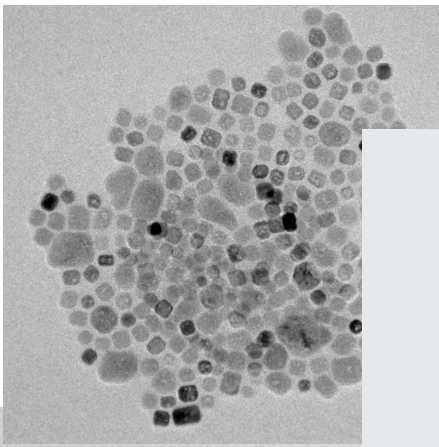
p-value =0.990273

Mean size increases with temperature in low  $[\text{MgCl}_2]$ , however, at low  $[\text{MgCl}_2]$  it diminishes



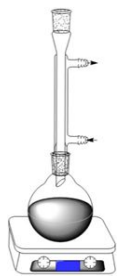
Further details on statistical analysis visit poster: XXXXXX

All the tested  
experimental conditions  
yield Nps with  
appropriate size for the  
application in PDT



# Ligand exchange

OA for PVP

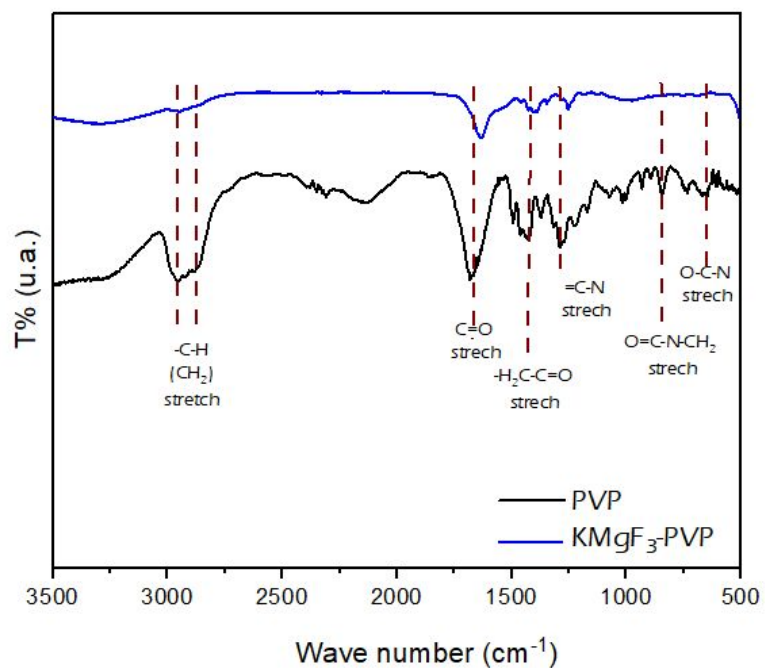


KMgF<sub>3</sub>-AO NPs

Ethyl acetate:Ethanol 2:1

Polyvinylpyrrolidone (PVP)

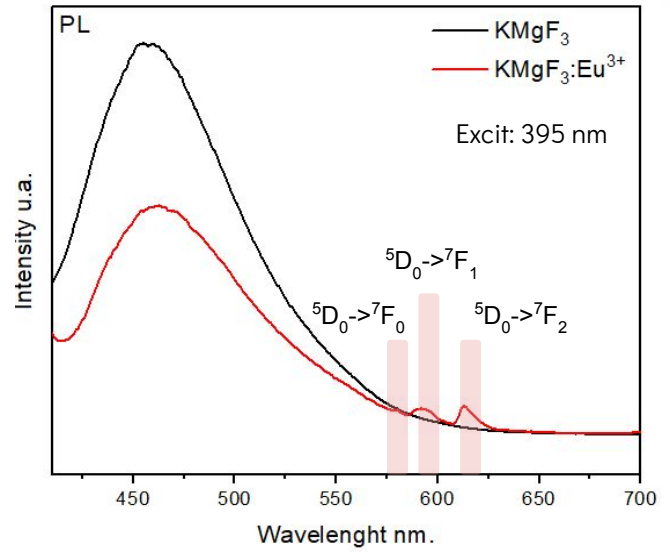
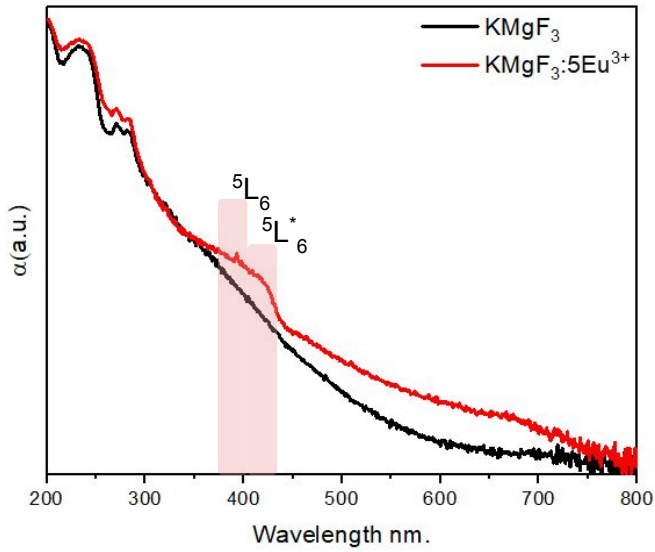
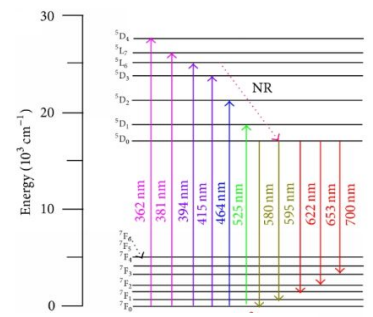
130°C- 4h Reflux



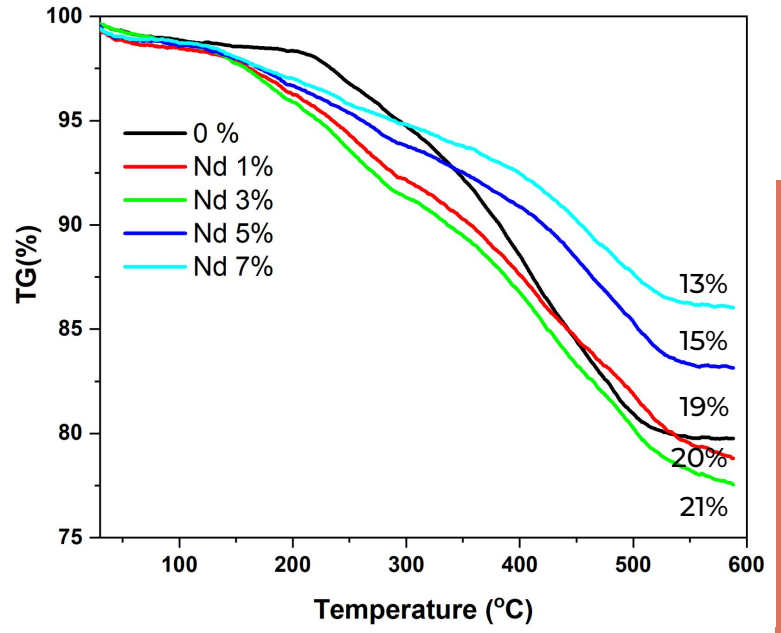
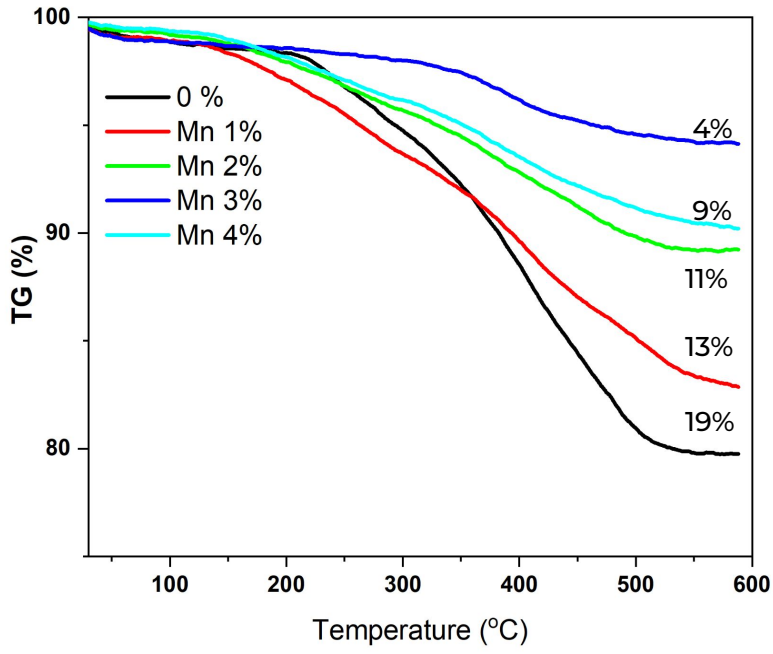
# KMgF<sub>3</sub> doping - Eu<sup>3+</sup>



Eu(NO<sub>3</sub>)<sub>3</sub> precursor

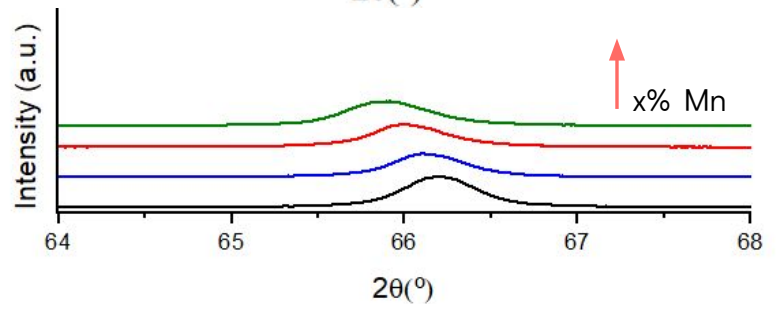
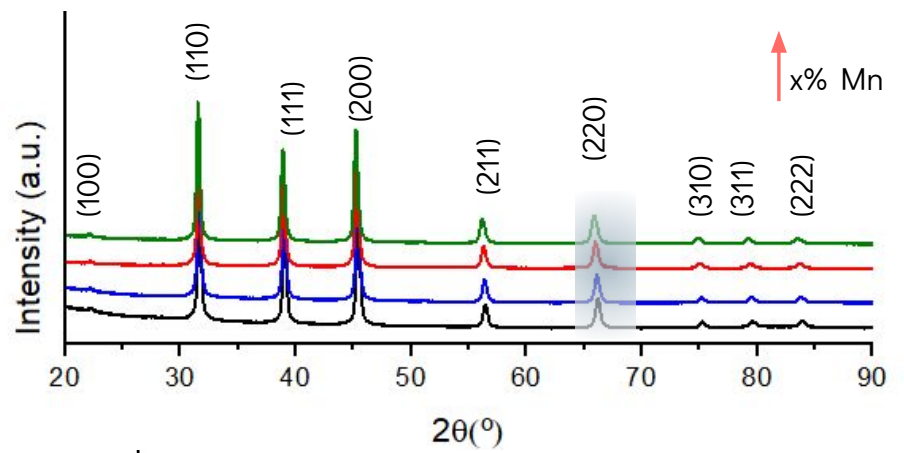


# TG results

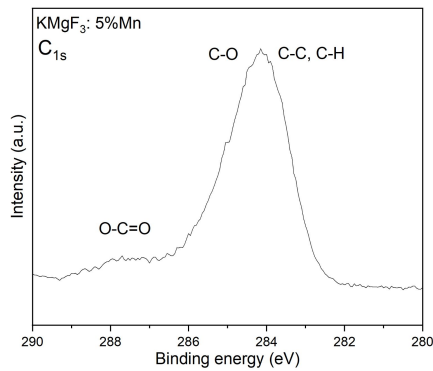
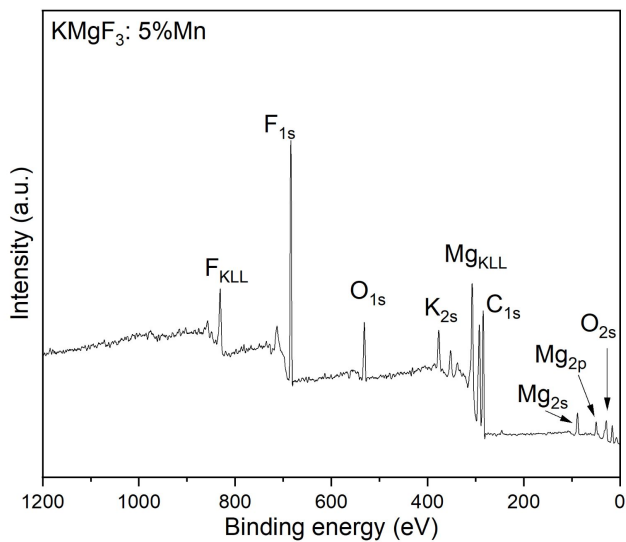


# KMgF<sub>3</sub> doping - Mn<sup>2+</sup>

KMgF<sub>3</sub> unique phase  
Shift to smaller angles

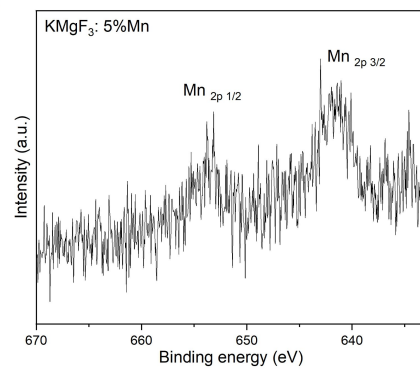


# XPS results



Surface composition (at %):

C= 43.2%; O=7.7%, F=17.2%, K=11.8%, Mg= 19.9%, Mn < 1%

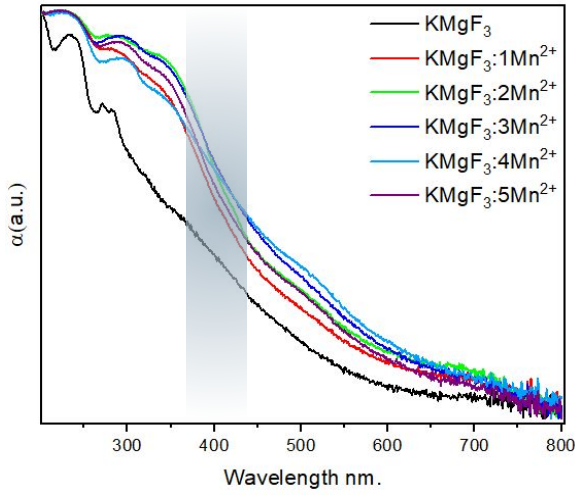




# KMgF<sub>3</sub> doping - Mn<sup>2+</sup>

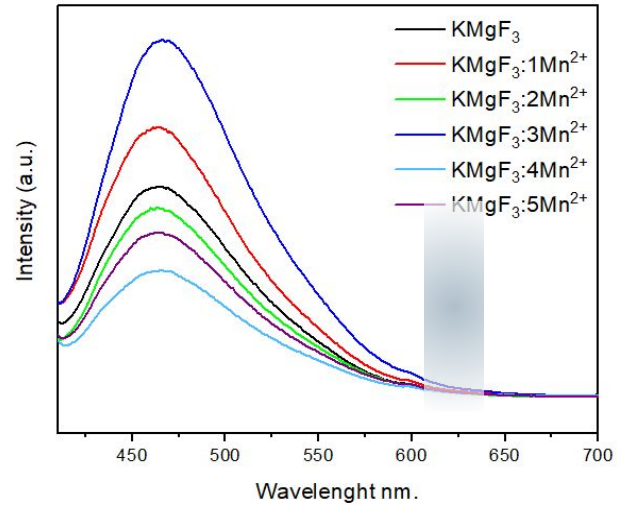
KMg<sub>1-x</sub>F<sub>3</sub>:xMn<sup>2+</sup> (x= 0.1,0.2,0.3,0.4,0.5)

C<sub>4</sub>H<sub>6</sub>MnO<sub>4</sub> precursor

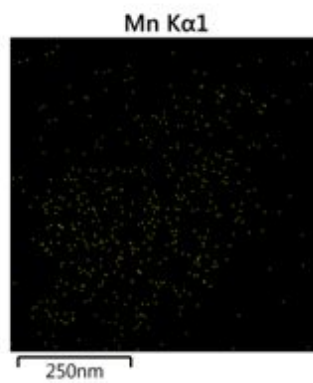
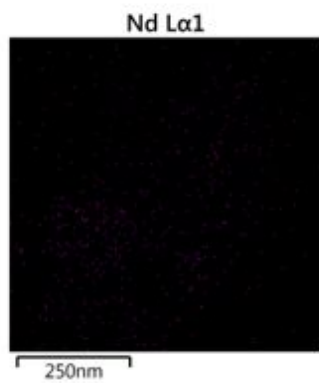
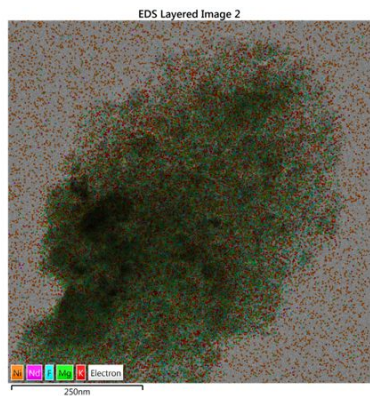
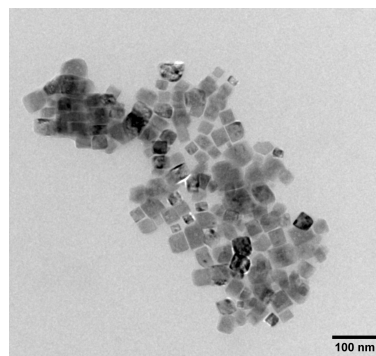


Expected a band around 400 nm

Expected a band around 600 nm

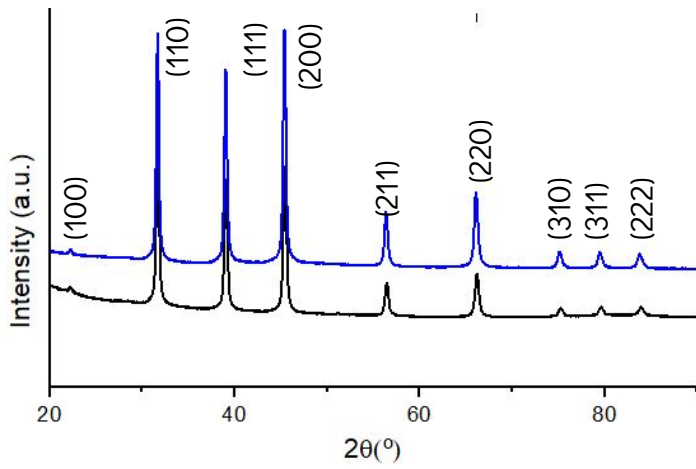


# KMgF<sub>3</sub> doping - Mn<sup>2+</sup> 4%

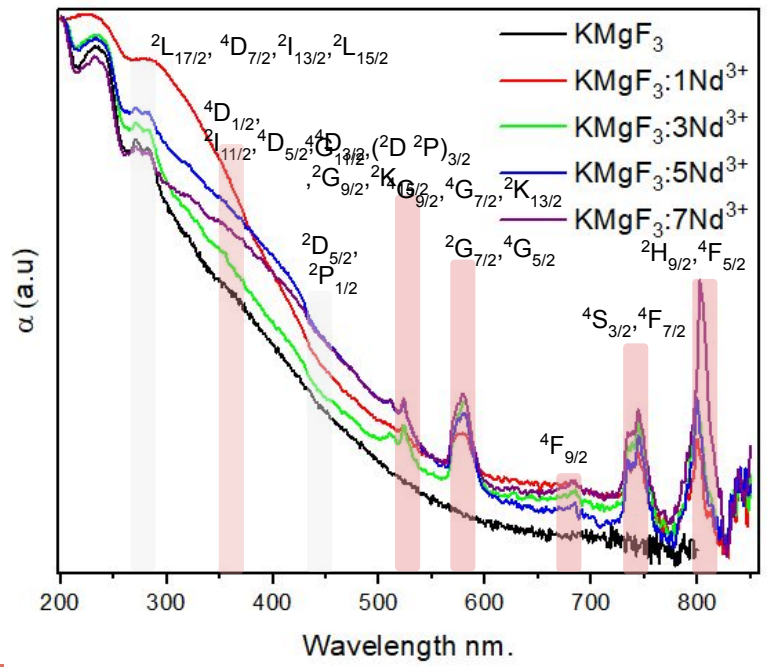


# KMgF<sub>3</sub> doping - Nd<sup>3+</sup>

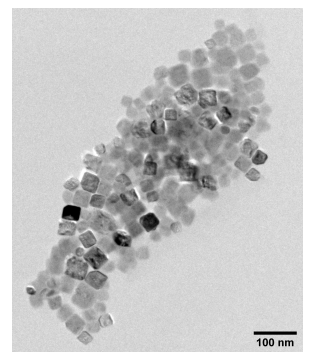
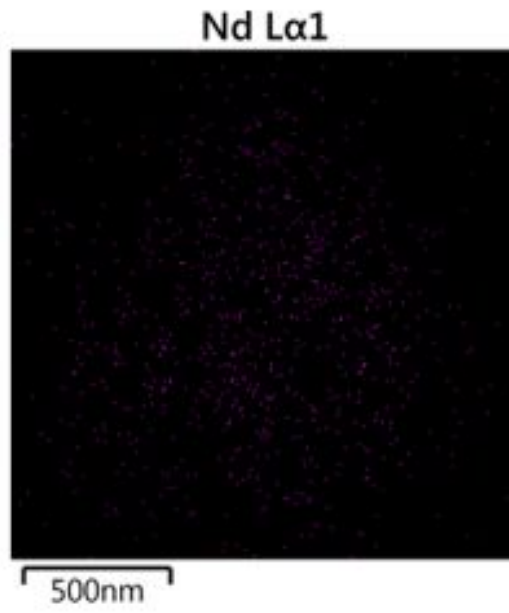
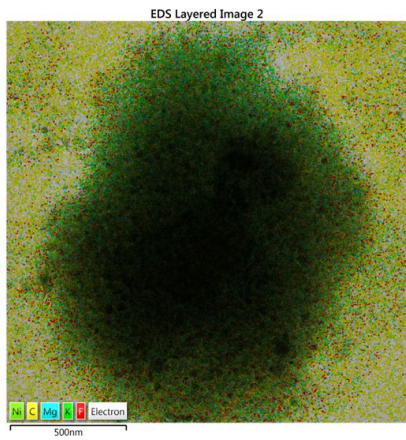
KMgF<sub>3</sub>:xNd<sup>2+</sup> (x=0.1, 0.3, 0.5, 0.7)



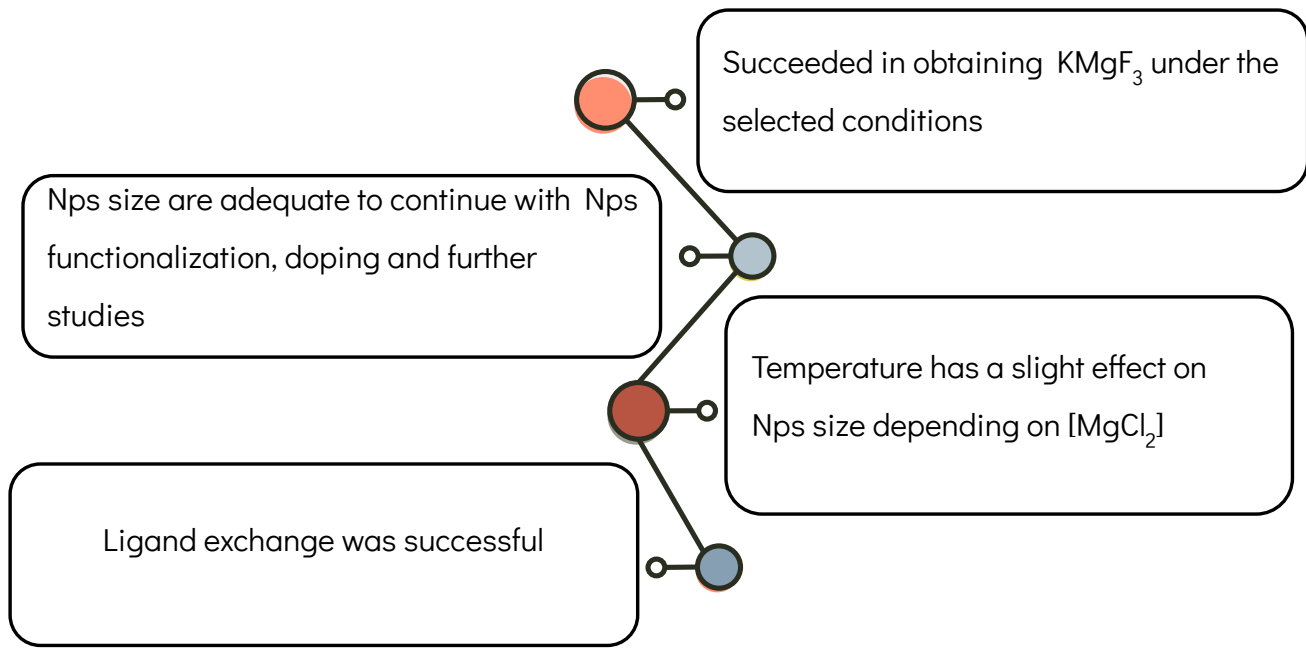
No impurities were found when Nd<sup>3+</sup> was added



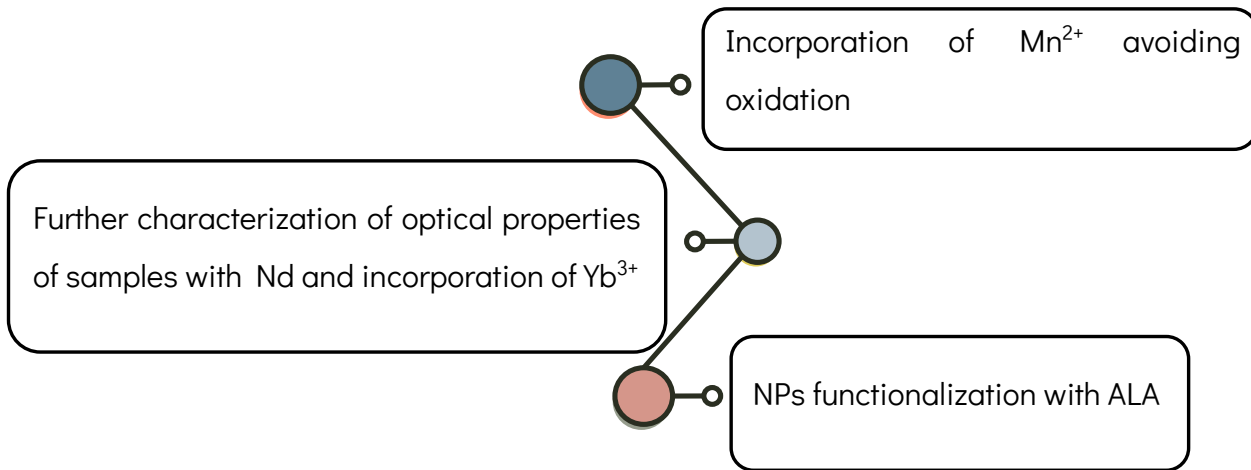
# KMgF<sub>3</sub> doping - Nd<sup>3+</sup>



# Up to now



# Working in progress...



# NANOMATERIALES

## NANOMEDICINA

Lic. Romina Keuchkerián (FQ)  
Dr. Mauricio Rodríguez (CURE)  
Dr. Wilner Martínez (IIBCE)  
Dra. Ivana Aguiar (FQ)  
Msc. Isabel Galain (FQ)  
Dra. Carolina Crisci (CURE)  
Dr. Leopoldo Suescun (FQ)  
Bqco. Alvaro Olivera (CURE)  
Tec. Heinkel Bentos Pereira (CURE)

# FINANCIACIONES





**THANK YOU**

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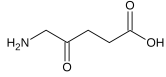
[meperez@cure.edu.uy](mailto:meperez@cure.edu.uy)

# Terapia fotodinámica

## Nanopartículas de $\text{KMgF}_3$



$\text{KMgF}_3$ :Nd: Yb:Mn



Fotosensibilizador



Nd: Activación a 808 nm



Ventana biológica

- Tesis de Maestría en Qca en curso "Desarrollo de nanopartículas para aplicación en terapia fotodinámica", Romina Keuchkerian
- Proyecto FCE II "Desarrollo de nanopartículas con propiedad de conversión ascendente para potenciales aplicaciones Biomédicas" FCE-1-2020-162287, resp. Romina Keuchkerian

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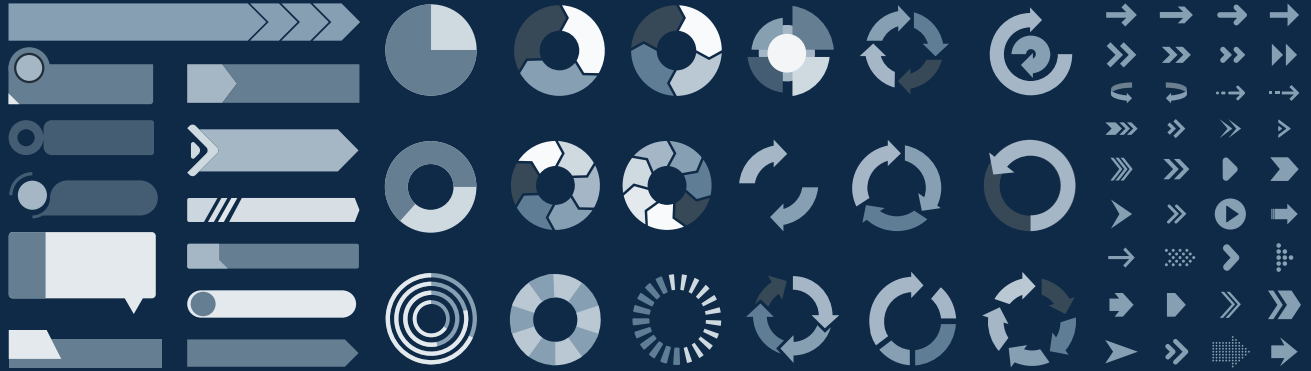
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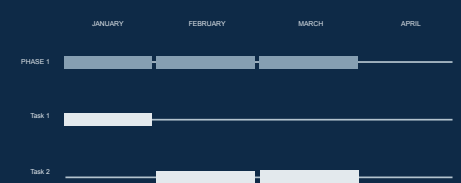
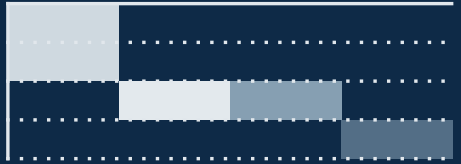
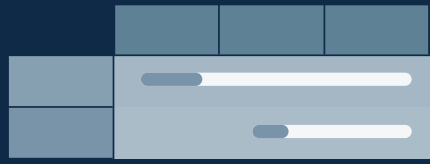
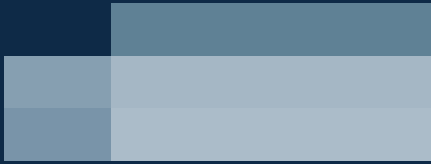
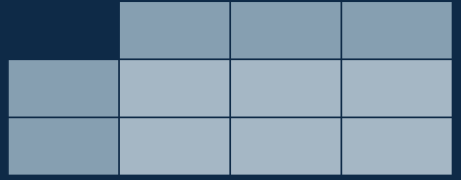
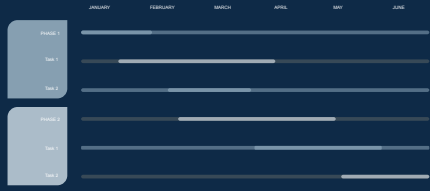
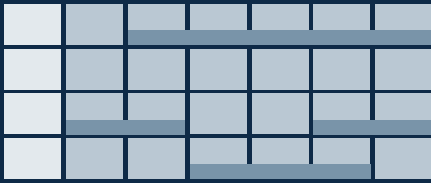
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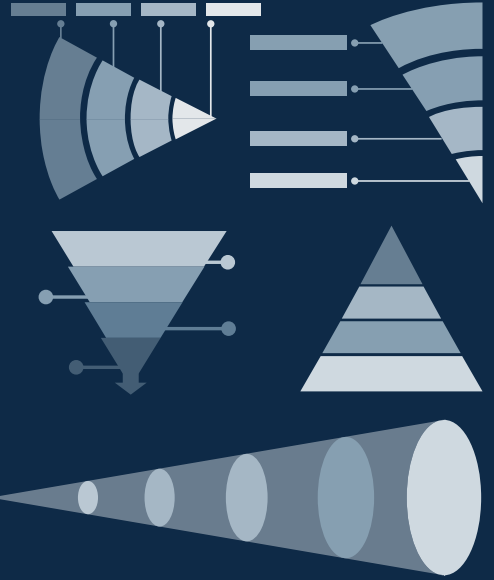
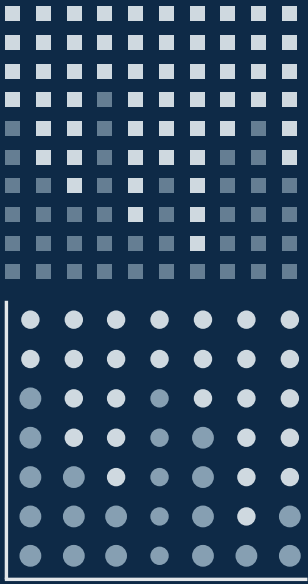












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## Educational Icons



## Medical Icons



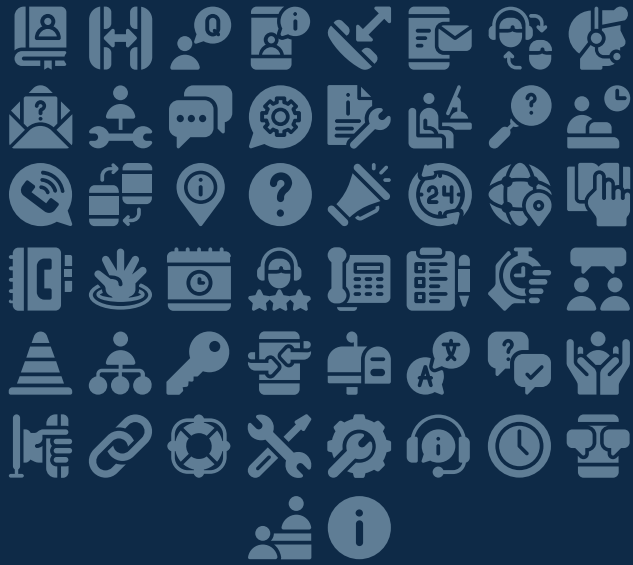
## Business Icons



## Teamwork Icons



## Help & Support Icons



## Avatar Icons



## Creative Process Icons



## Performing Arts Icons





# Nature Icons

