PolyChar 23

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# From loaded shell-core microcapsules to thermoplastic hybrid composites: A new pathway for the preparation of conductive and magnetic polyamide composites

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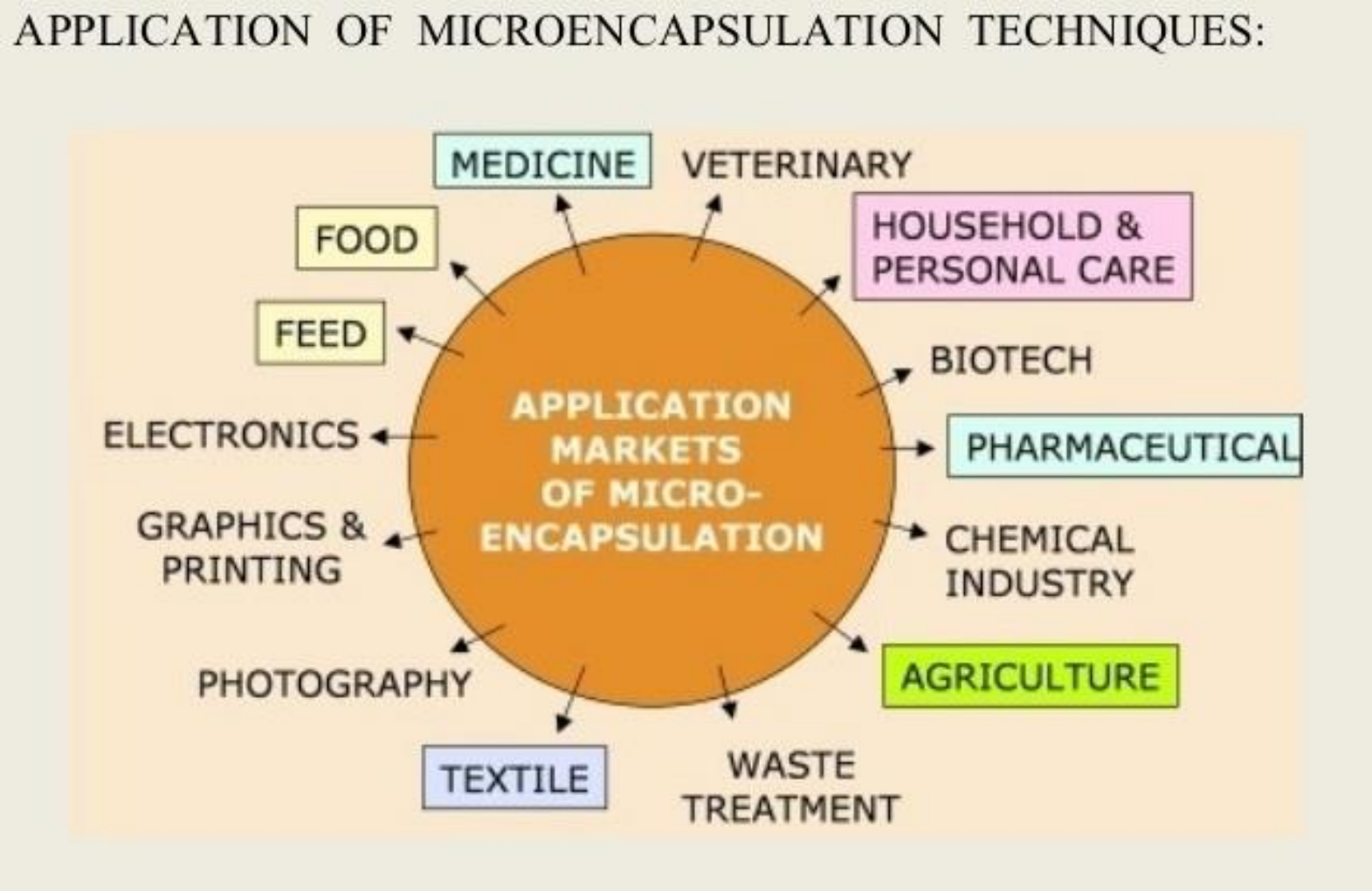
POLYCHAR 23

IUPAC
INTERNATIONAL UNION OF PURE AND APPLIED CHEMISTRY

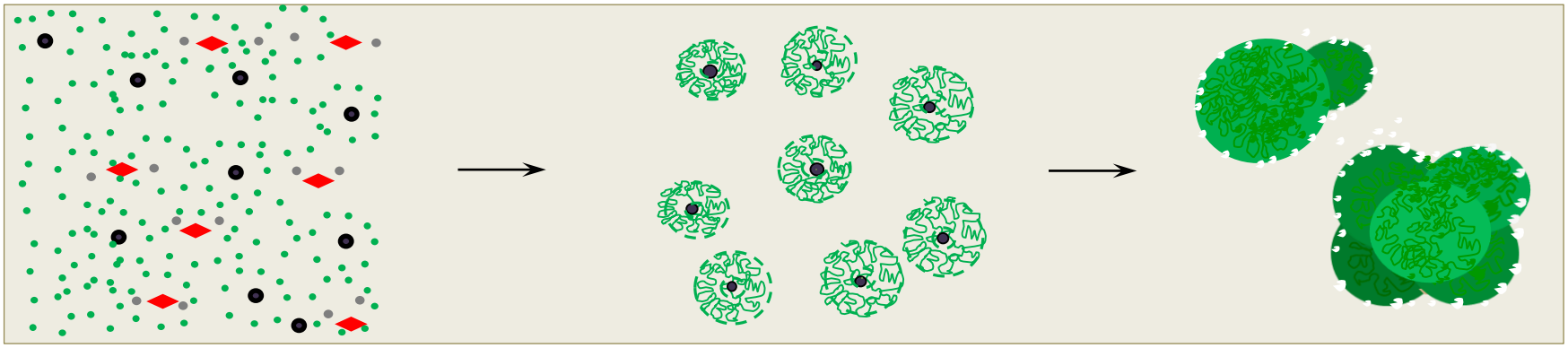
# Outline

- Motivation
- One-pot synthesis of polyamide 6 microcapsules with different payloads
- Transforming PA6 microcapsules into hybrid composite materials and their mechanical, electroconductive and magnetic properties
- Conclusions

# Microencapsulation – applications



# One-pot synthesis of PAMC



**Starting mixture**

- *Monomer*
- *Catalytic system*
- *Payload*
- *Solvent*

**Viscous particles**

**Microcapsules**

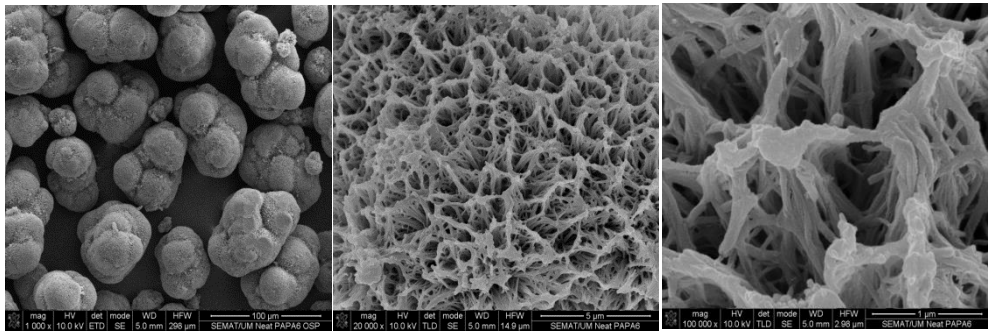
**Multifaceted applications**

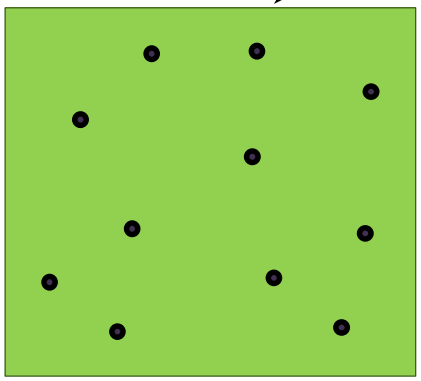
***CM, IM***

**Payloads used (1-10 wt.%):**

- *Natural nanoclays, synthetic Al/Ti sillicates*
- *Carbon allotropes – CB, CNT, CNF, Fullerenes*
- *Metal & Metal oxides – Cu, Al, Mg, Fe, $Fe_3O_4$*

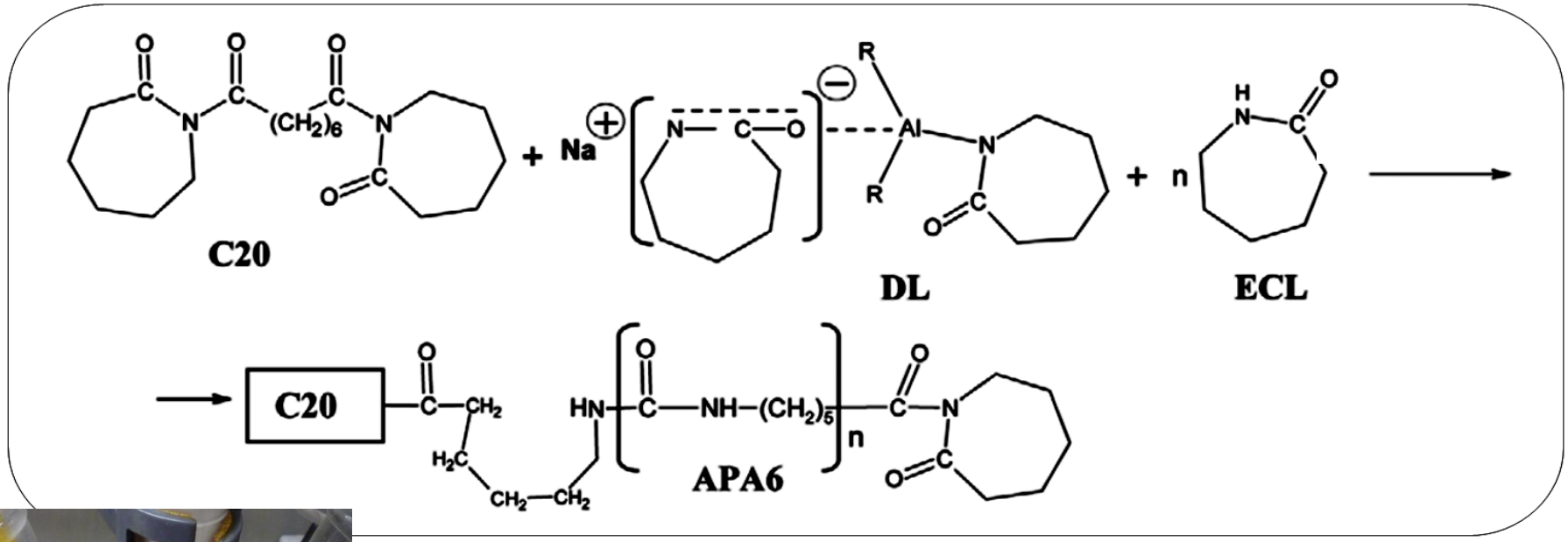
**PAMC**





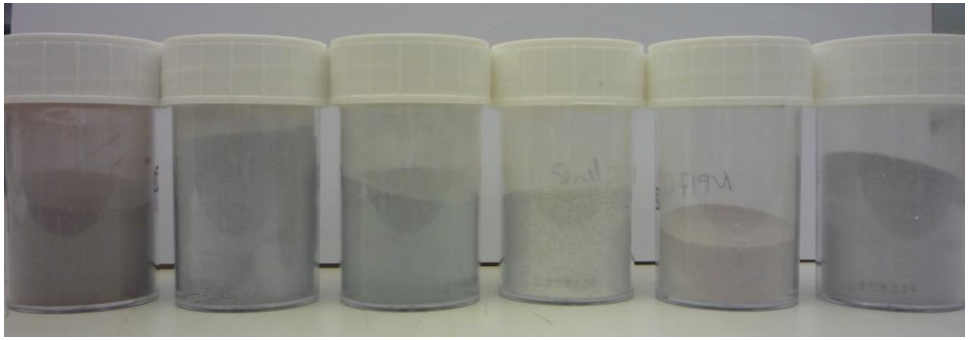
**Hybrid composite materials with electro-conductive & magnetic properties**

# One-pot synthesis of PAMC – reaction




## Payloads used (1-10 wt.%):

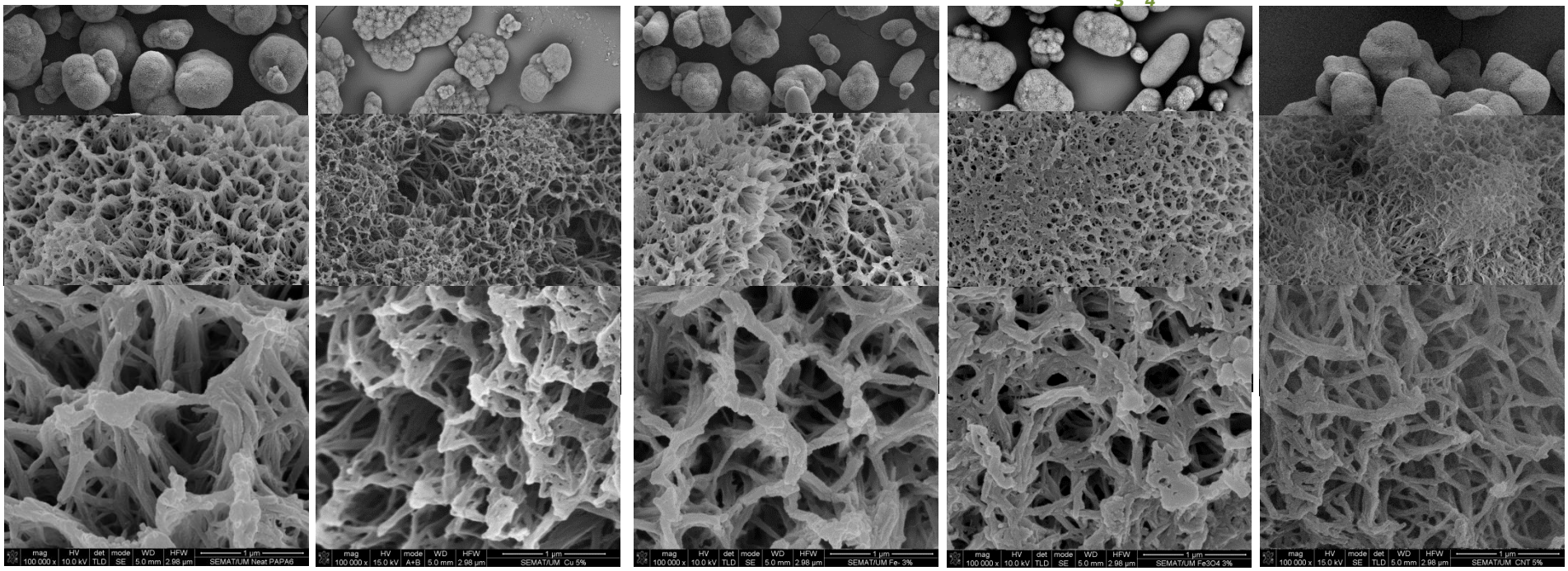
- *Natural nano-clays (CLOISITE 15A, CLOISITE 20A );*
- *Synthetic Al/Ti sillicates*
- *Carbon allotropes – CB, CNT, CNF, Fullerenes*
- *Metal & Metal oxides – Cu, Al, Mg, Fe, $Fe_3O_4$*



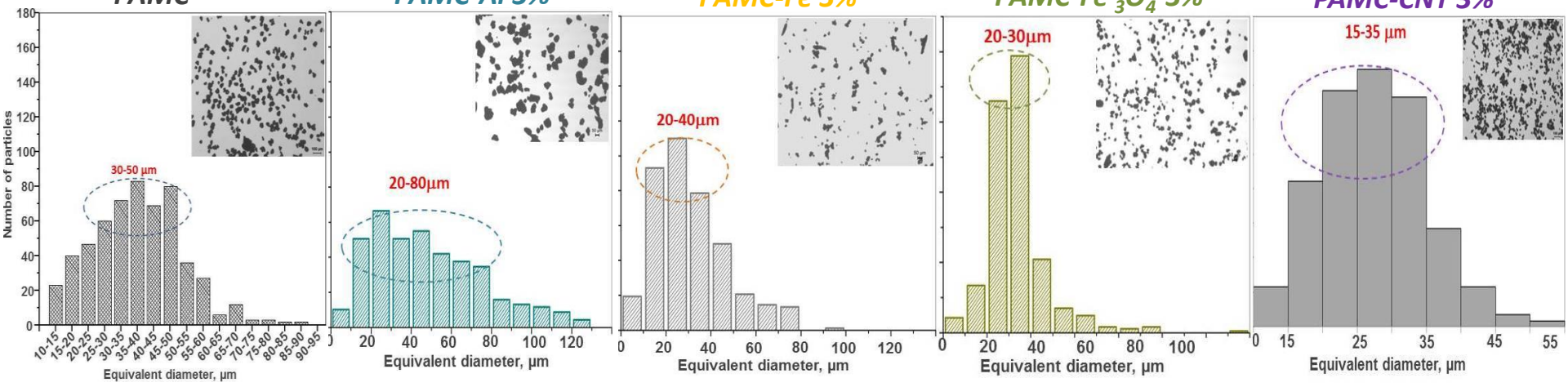
*Pat. Appl. 12345/2015*

# PAMC - Morphology

PAMC | PAMC-Al 3% | PAMC-Fe 3% | PAMC-Fe $_3O_4$-3% | PAMC-CNT 3%

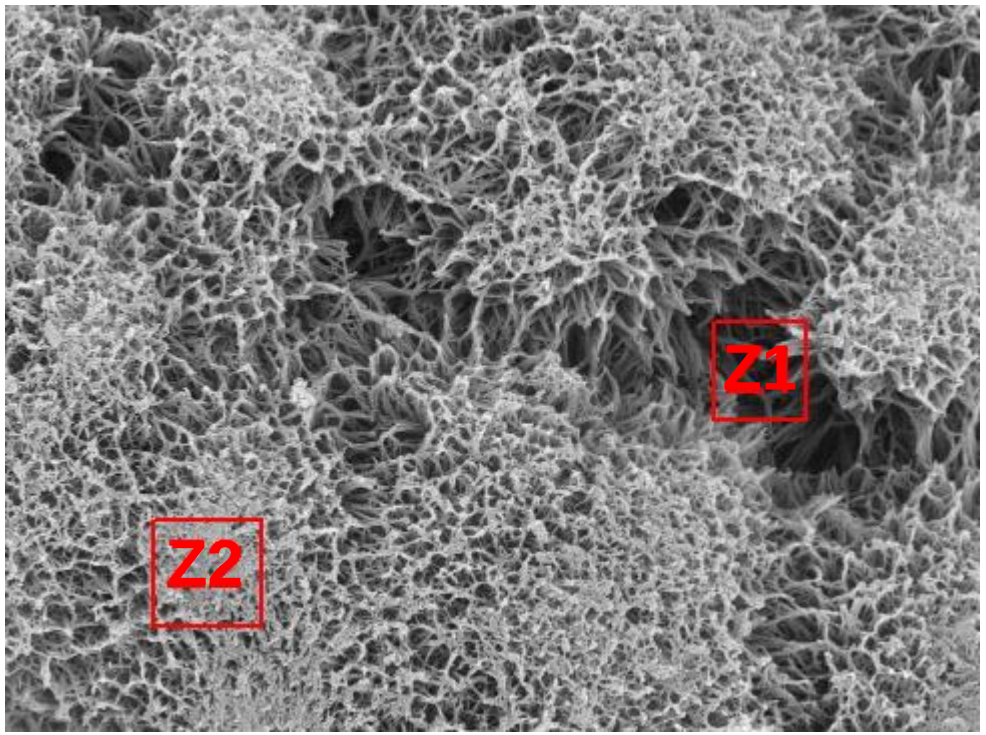


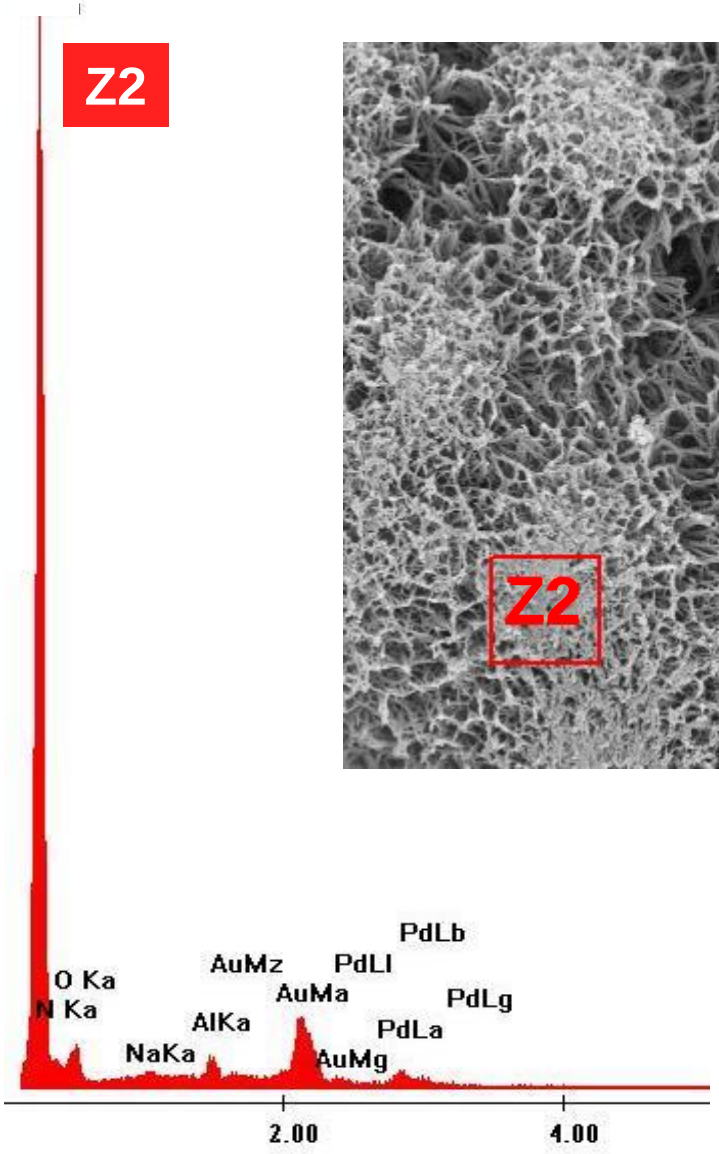
## Granulometric analysis

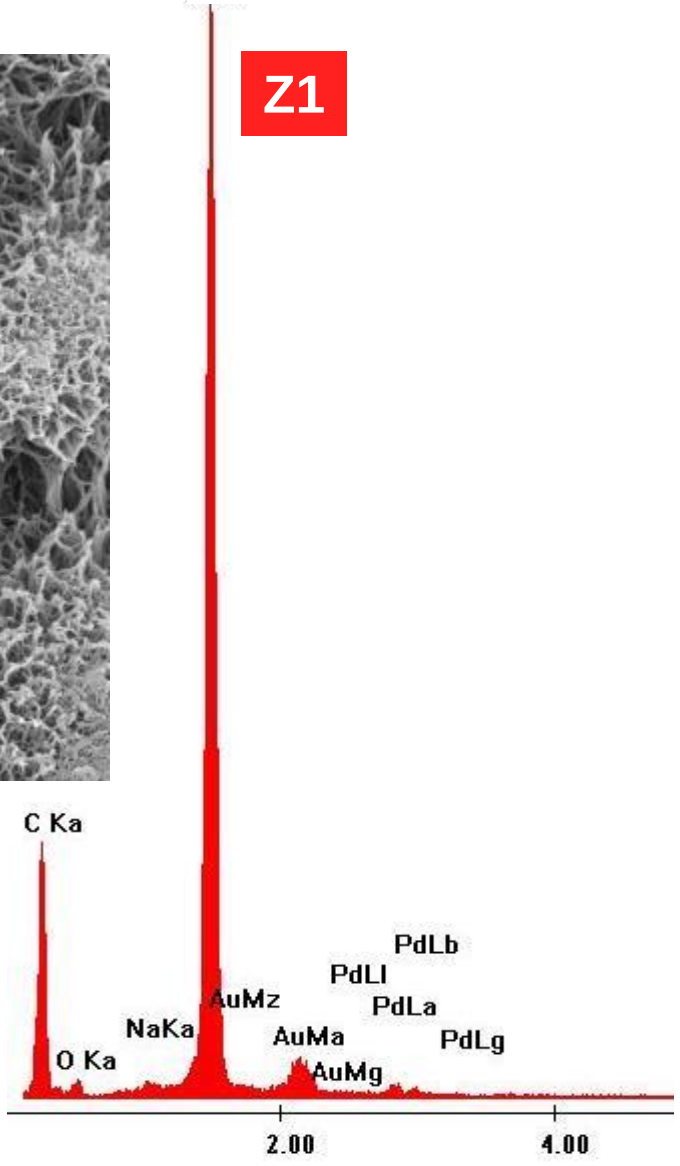


# PAMC – Core-shell structure

## *SEM + EDX of PAMC-Al 3%*

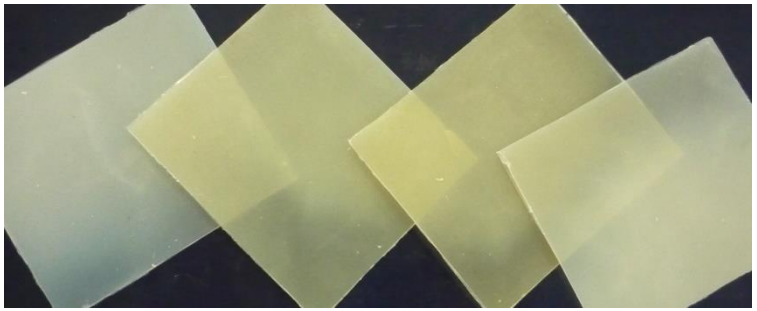


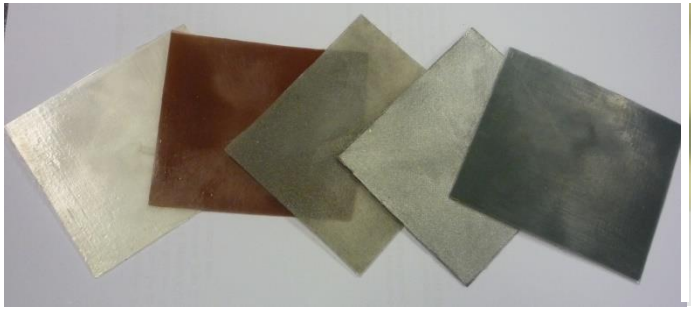



# Hybrid composites by CM of PAMC

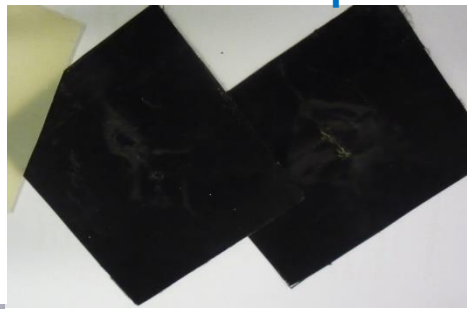
## APA6-Cloisites

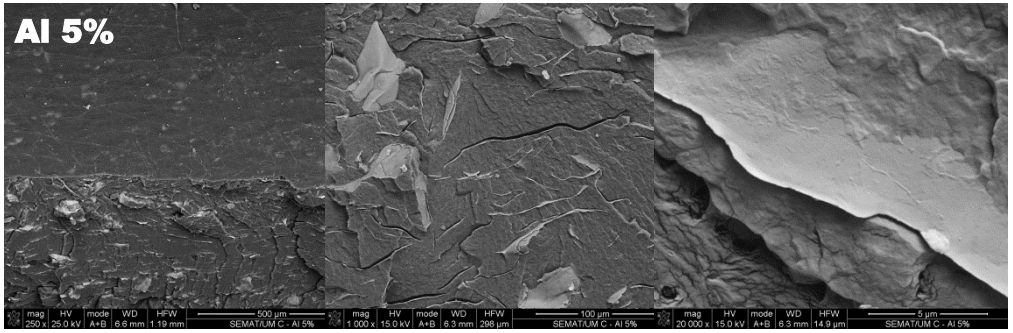


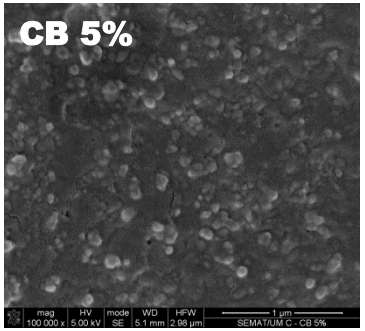
## APA6-Me&MeO



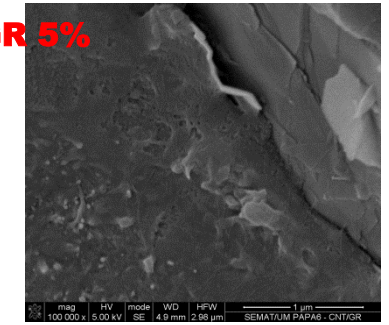
## APA6-C allotropes

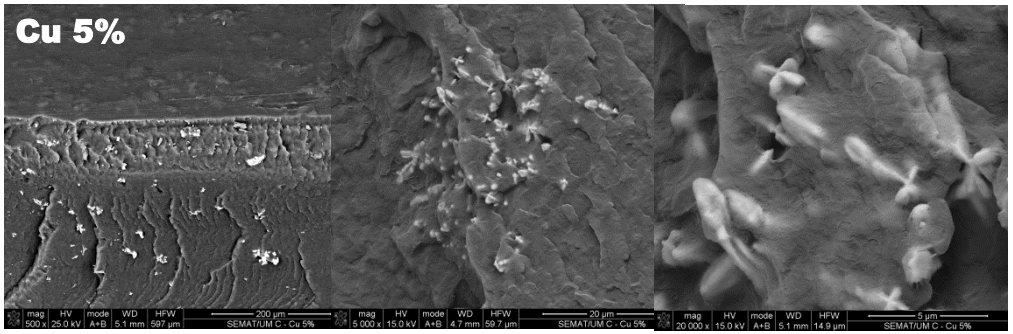


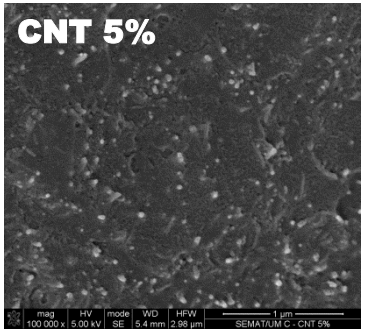


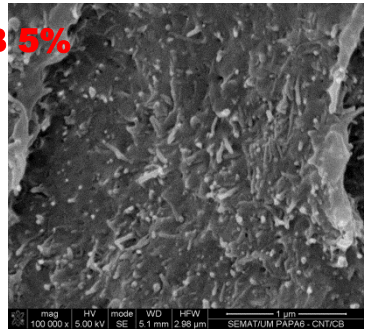

CNT/GR 5%

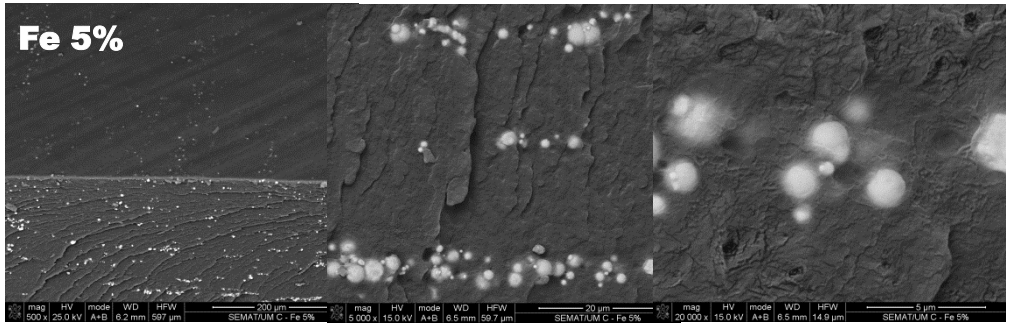


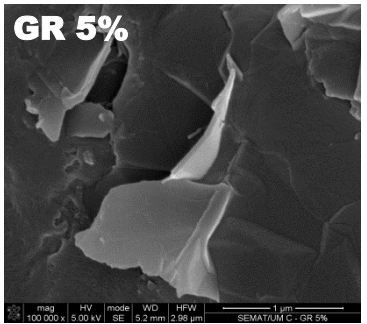


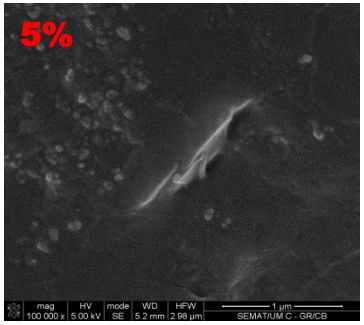

CNT/CB 5%





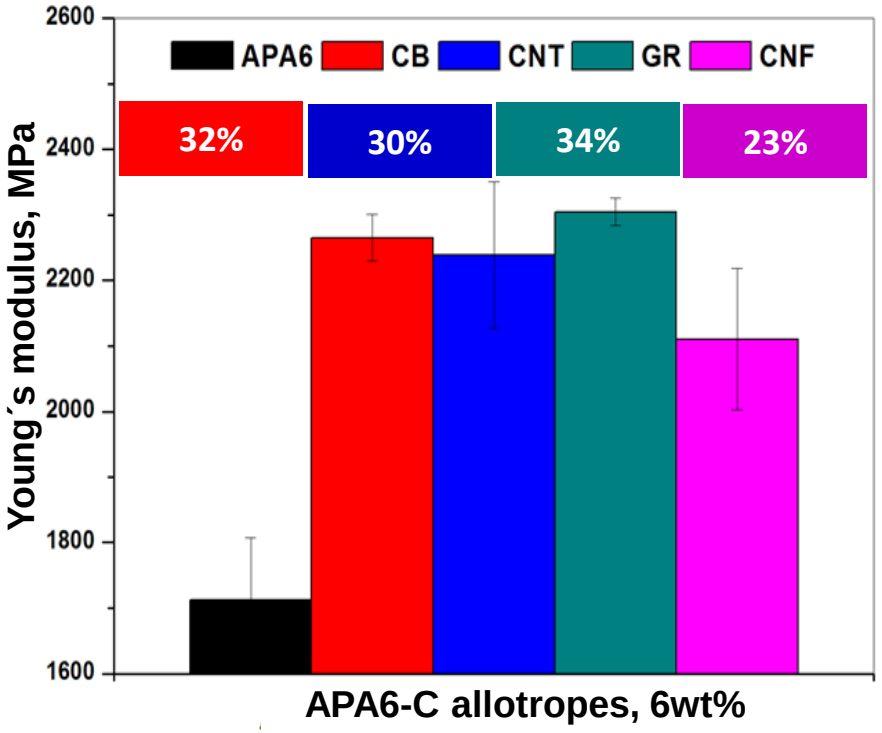




GR/CB 5%

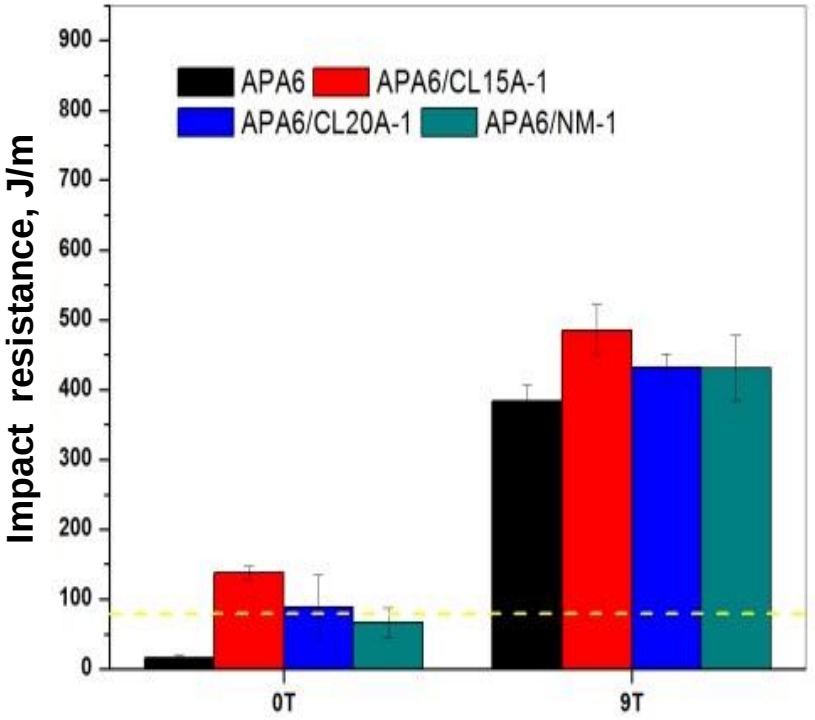


# Mechanical properties of some composites prepared with PAMC

## Tensile moduli of PA6/C allotropes composites




## Impact resistance of PA6/PA66 laminates




**PA6**
**1,5 GPa/70 MPa**

**Young´s moduli**
**12-14 GPa**

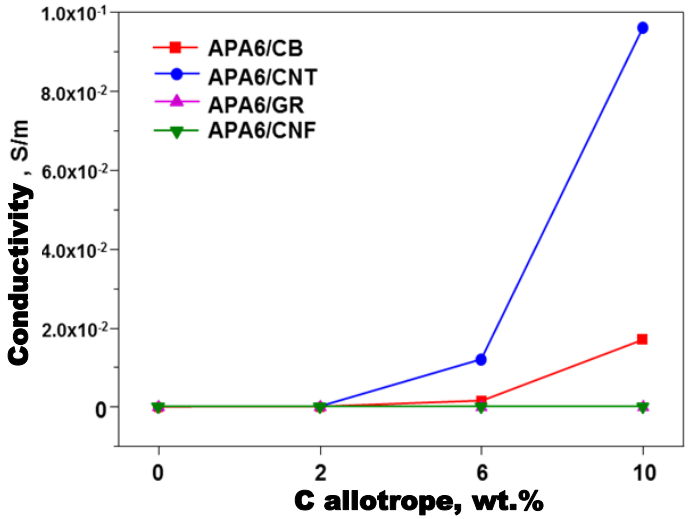
**Tensile strengths**
**410 – 520 MPa**

## Tensile properties of PA6/ GF Textile laminates prepared with PAMC

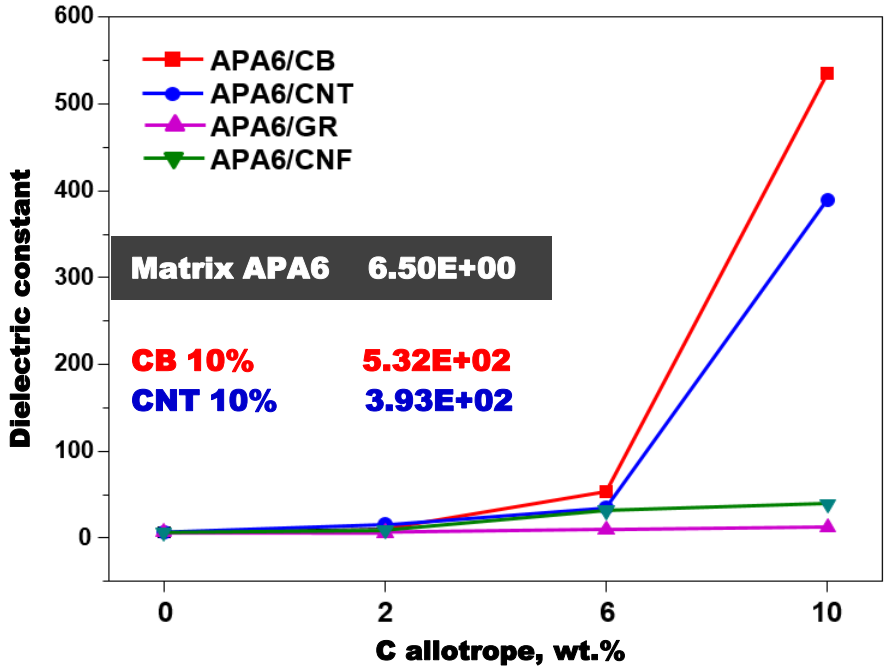
| Number of layers | Sample | Young's modulus, E [GPa] | Tensile strength, $\sigma_{máx}$ [MPa] | Strain at break, $\varepsilon_{rot}$ [%] |
|---|---|---|---|---|
| 10 | **APA6-10T(FV1)** | 12.34±0.35 | 411.2±28.4 | 6.56±0.6 |
| | **APA6-10T(FV1)*** | 13.97±0.09 | 497.2±30.6 | 6.4±0.5 |
| | **APA6-10T(FV2)*** | 14.14±0.77 | 509.1±23.9 | 7±0.6 |
| | **APA6/CL20A1-10T(FV1)** | 13.36±1.01 | 482.1±21.5 | 6.5±0.7 |
| | **APA6/CL20A1-10T(FV1)*** | 12.23±0.73 | 416.7±40.9 | 5.7±0.4 |

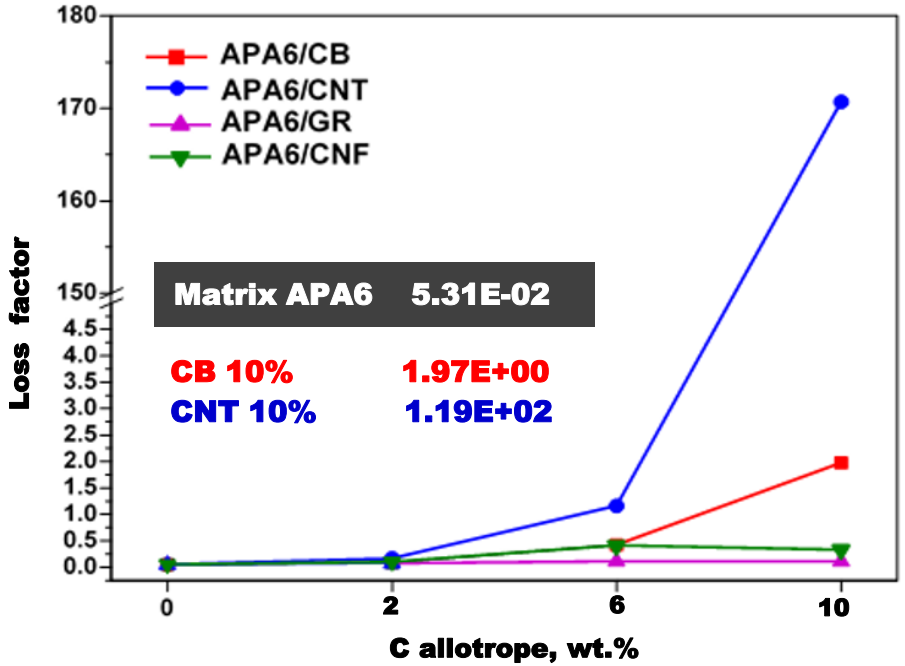
* Matriz com $Sb_2O_3$

# Electro-conductive properties of PA6/C hybrides

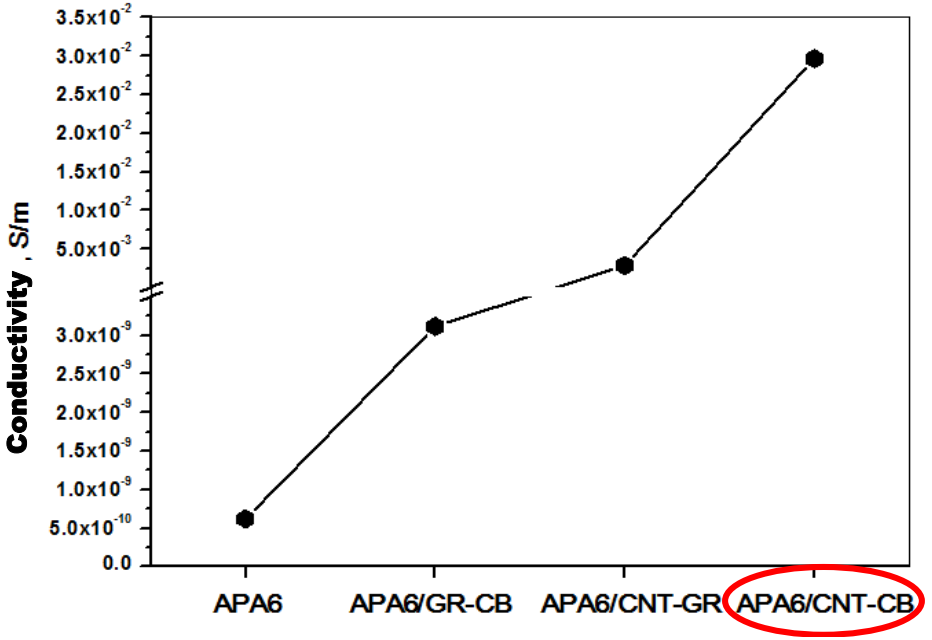


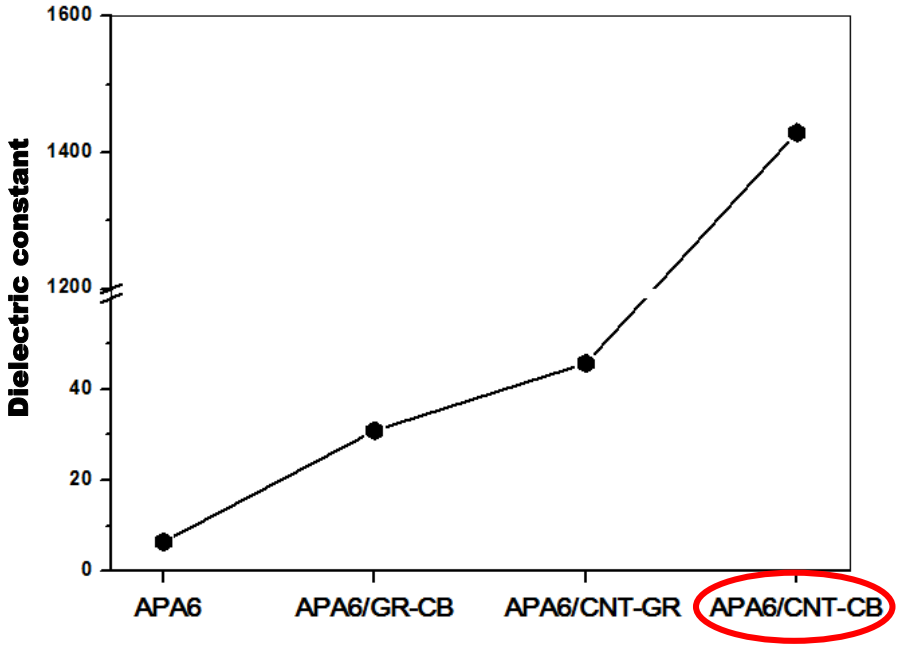

| Payload wt.% | 0 | 2 | 6 | 10 |
|---|---|---|---|---|
| CB | 6.2E-10 Isolating matrix | 4.6E-09 | 1.5E-03 | 1.7E-02 |
| CNT | | 6.6E-05 | 1.2E-02 | 9.6E-02 |
| GR | | 6.0E-09 | 3.4E-09 | 3.5E-09 |
| CNF | | 3.1E-09 | 6.5E-06 | 1.1E-06 |

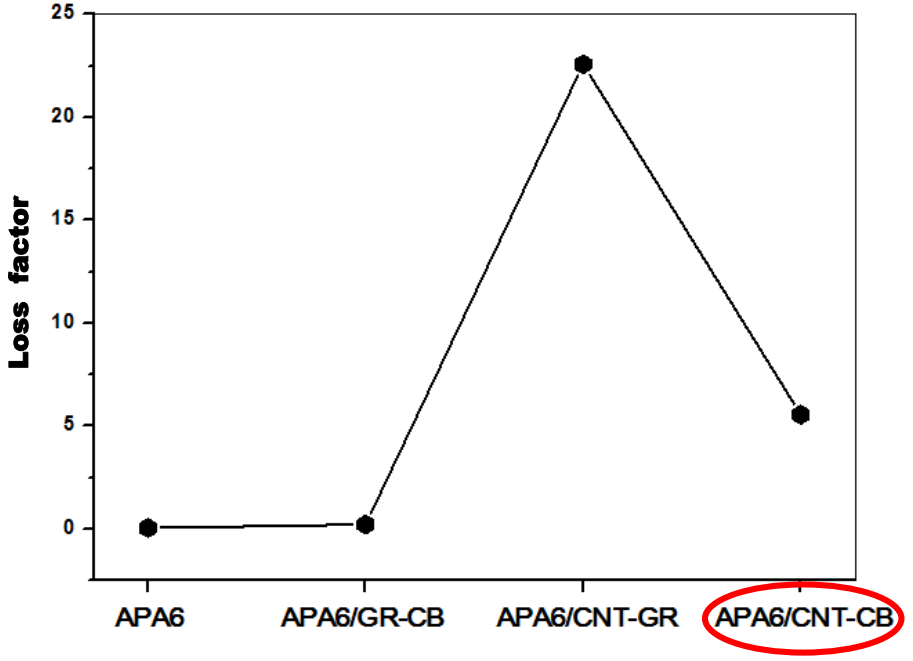


# Electro-conductive properties of PA6/mixed C allotropes




| Sample | σ, S/m | ε’ |
|---|---|---|
| APA6 | 6.2E-10 | 6.50E+00 |
| APA6/CNT-GR | 1.6E-02 | 4.80E+01 |
| APA6/CNT-CB | 8.9E-02 | 1.42E+03 |
| APA6/GR-CB | 3.1E-09 | 3.10E+01 |

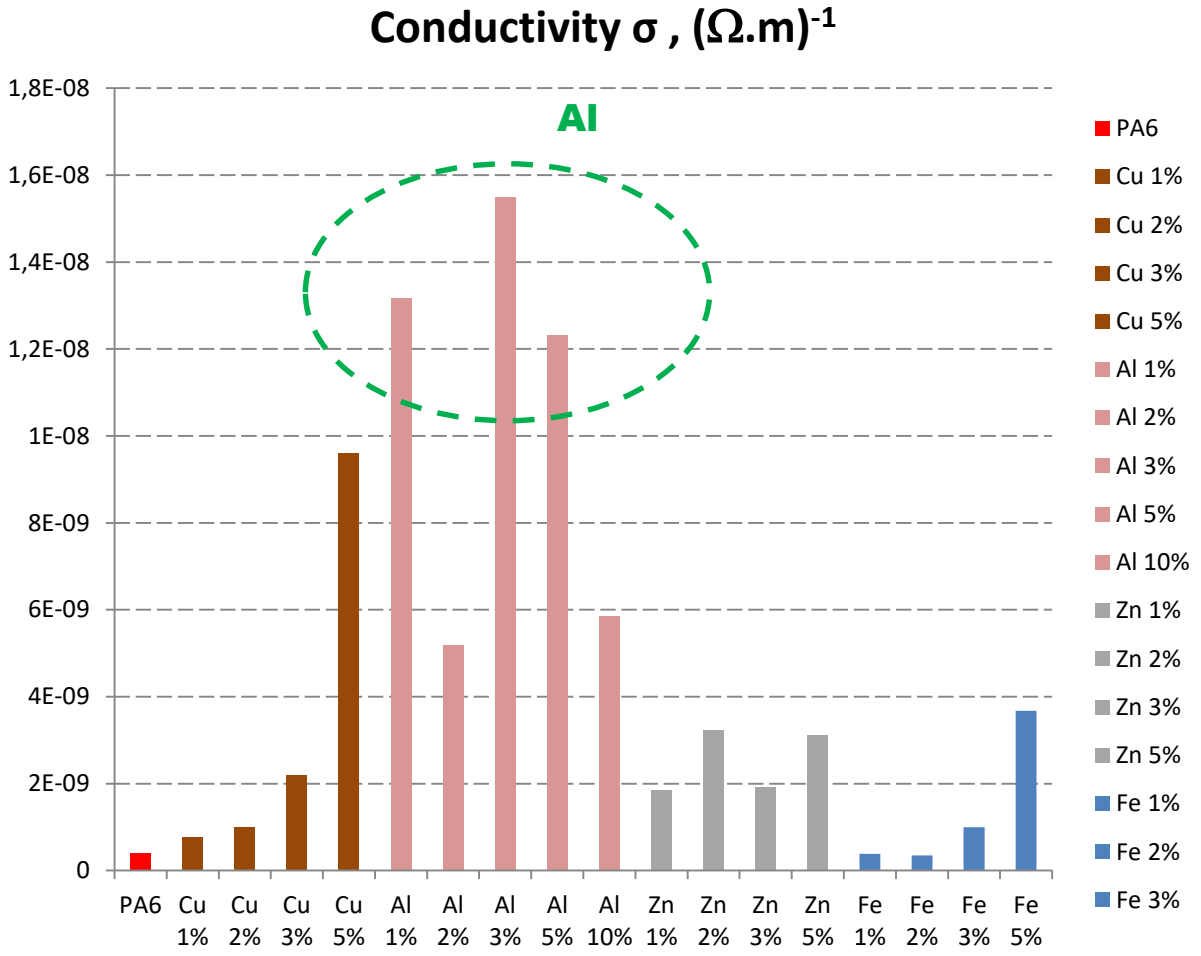
**APA6/CNT-CB**

σ 8.9E-02 S/m

ε’ 1.42E+03

tan δ 5.53E+00

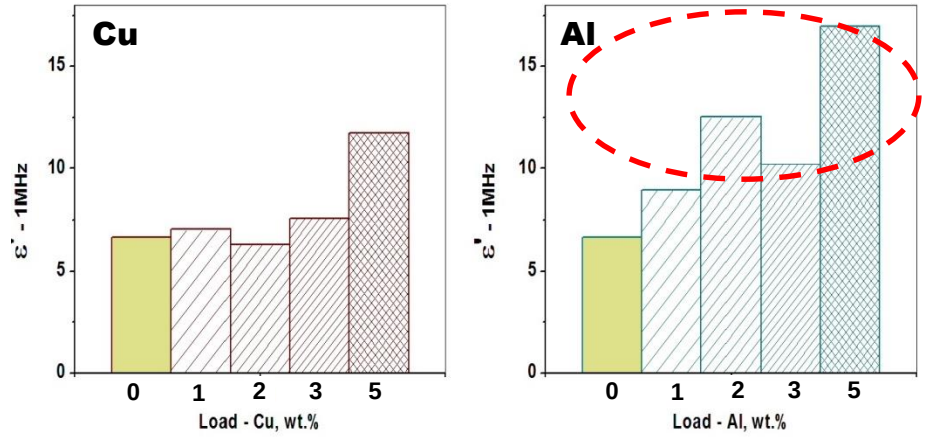
# Electro-conductive properties of PA6/Me hybrids




| Load, wt.% | PA6 | PA6-Cu | PA6-Al | PA6-Zn | PA6-Fe |
|---|---|---|---|---|---|
| 0% | 4,23E-10 | - | - | - | - |
| 1% | - | 8,56E-10 | 1,35E-08 | 1,76E-09 | 4,56E-10 |
| 2% | - | 1,05E-09 | 5,43E-09 | 4,51E-09 | 4,03E-10 |
| 3% | - | 2,20E-09 | 1,50E-08 | 1,85E-09 | 1,06E-09 |
| 5% | - | 9,42E-09 | 1,20E-08 | 3,45E-09 | 4,01E-09 |

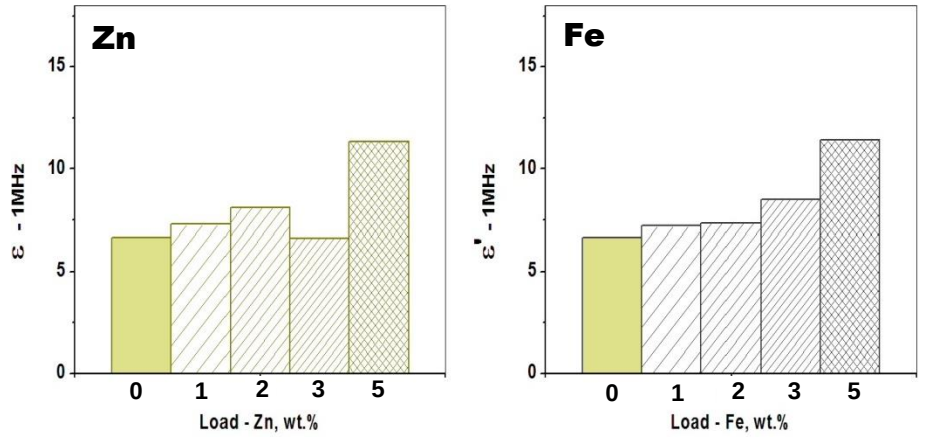
# Electro-conductive properties of PA6/Me hybrides

*Dielectric constant*





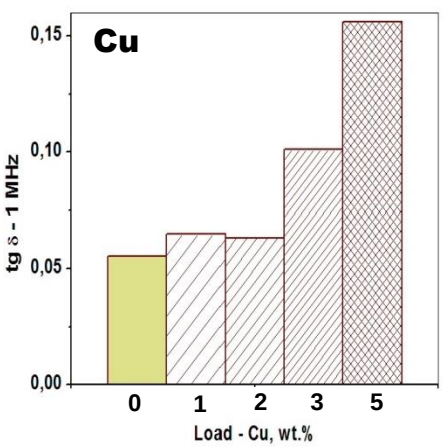


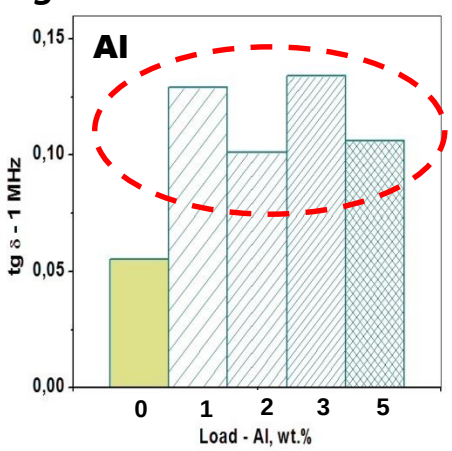


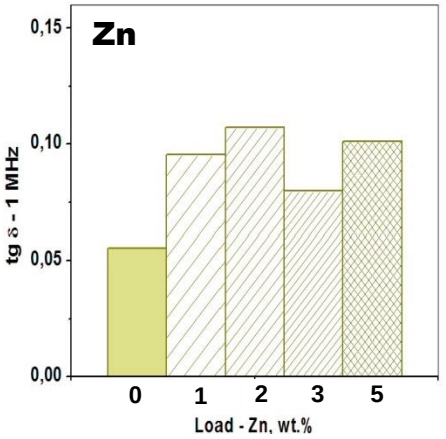


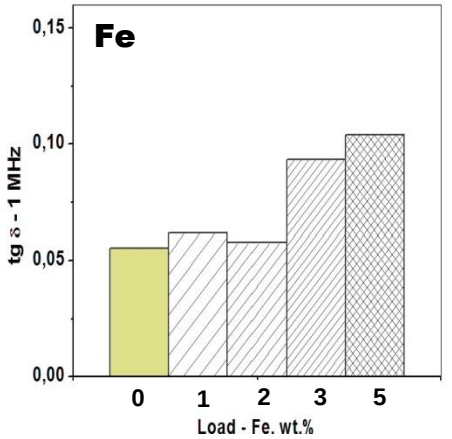


*Loss tangent δ*



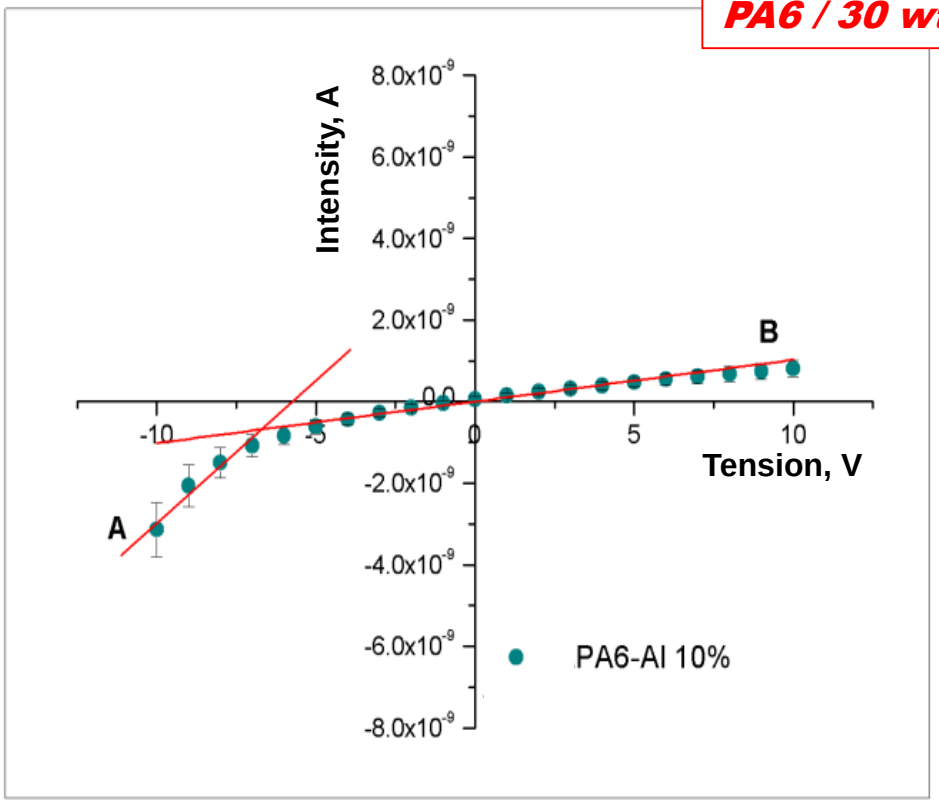



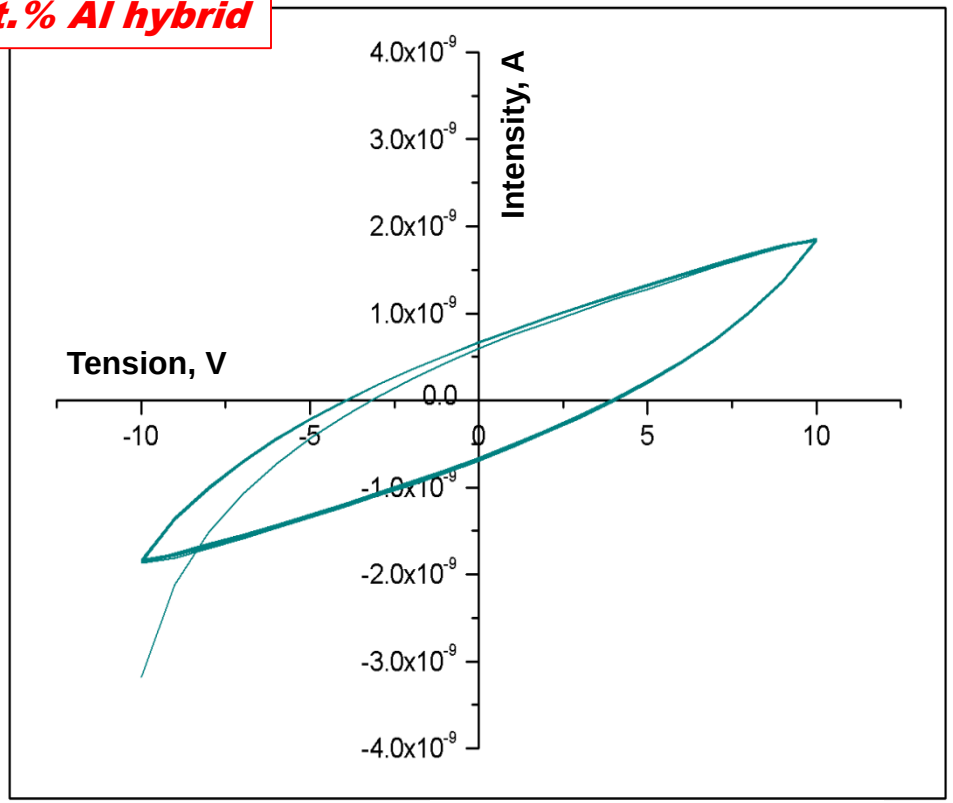



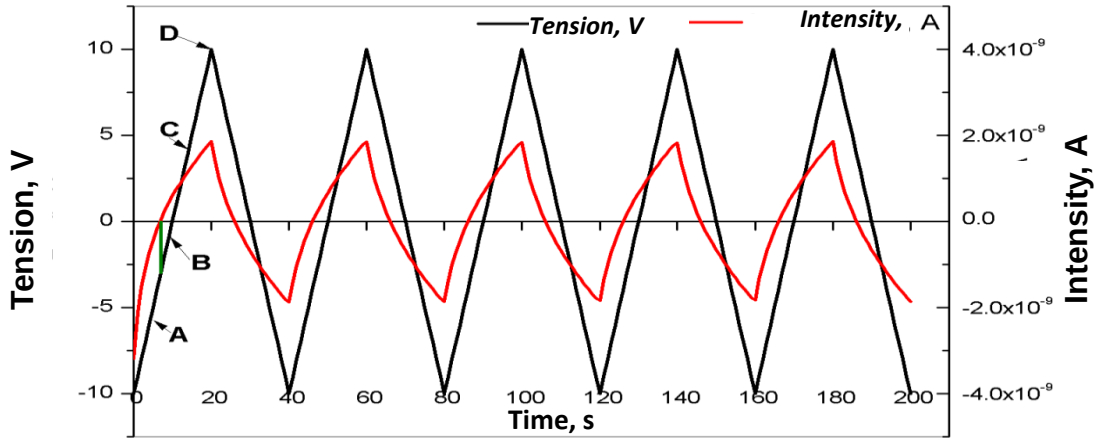

| Load, wt.% | PA6 | | PA6 - Cu | | PA6 - Al | | PA6 - Zn | | PA6 -Fe | |
|---|---|---|---|---|---|---|---|---|---|---|
| | ε' | tgδ | ε' | tgδ | ε' | tgδ | ε' | tgδ | ε' | tgδ |
| 0% | 6,78 | 0,052 | - | - | - | - | - | - | - | - |
| 1% | - | - | 7,13 | 0,066 | 8,77 | 0,134 | 7,49 | 0,100 | 7,56 | 0,075 |
| 2% | - | - | 6,32 | 0,063 | 11,22 | 0,095 | 8.17 | 0,096 | 7,33 | 0,059 |
| 3% | - | - | 7,60 | 0,101 | 12,38 | 0,128 | 6,62 | 0,080 | 8,29 | 0,093 |
| 5% | - | - | 10,30 | 0,128 | 16,55 | 0,114 | 9,10 | 0,091 | 10,82 | 0,095 |

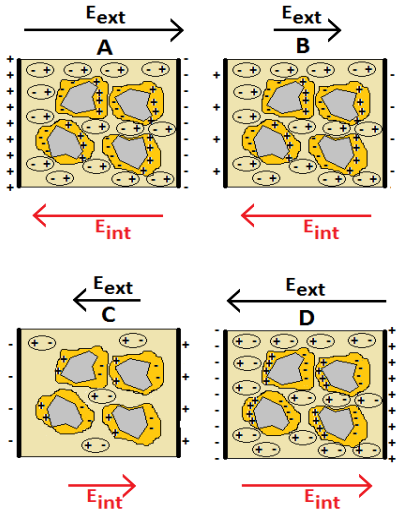
# Electro-conductive properties of PA6/10 % Al hybrid

***PA6 / 30 wt.% Al hybrid***

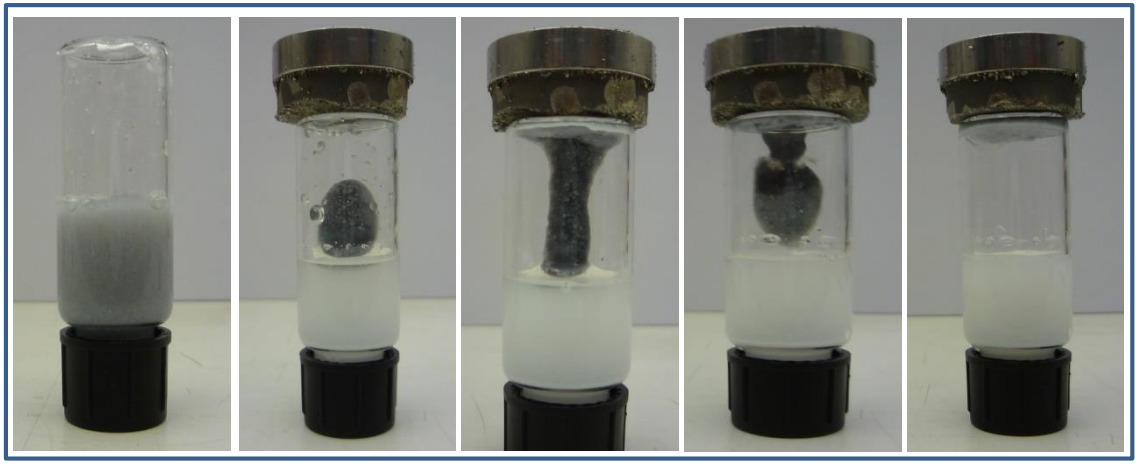


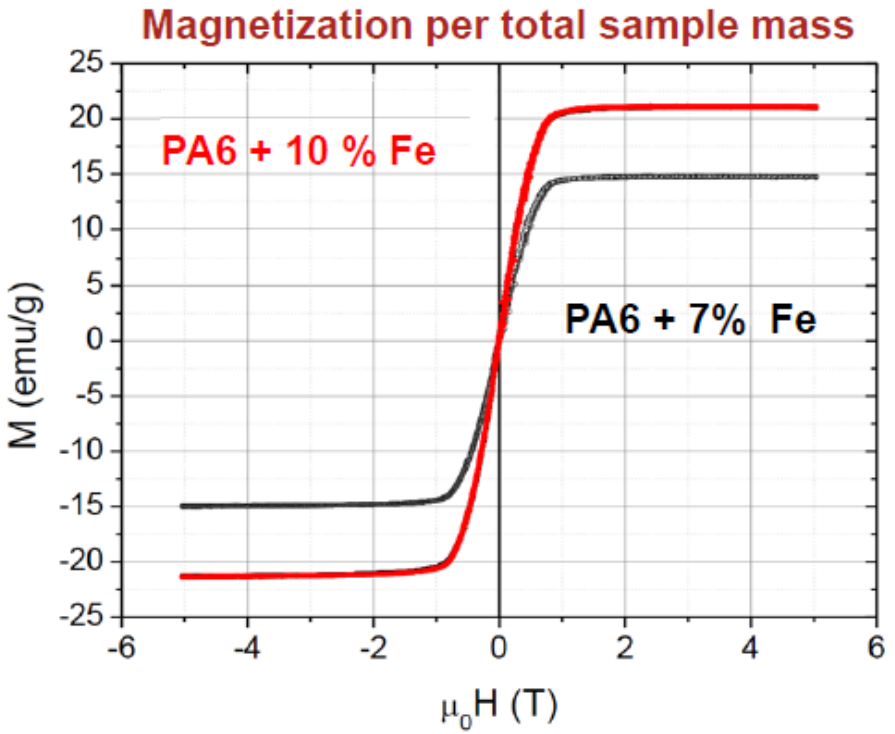



# Magnetic properties of PA6/Fe PAMC @ hybrides





# Conclusions

1. Polyamide hybrid microcapsules containing different loads can be prepared by an effective one-pot-synthetic method;

2. Using conventional processing technics these microcapsules can be transformed into hybrid composites with good load distribution resulting in tailored mechanical and electromagnetic properties.

3. PA6@Carbon allotrope composites thus prepared showed semi-conductor properties, while composites containing metal and metal oxide particles displayed properties that could be interesting for energy storage and energy absorbing materials.

**Acknowledgements:**

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