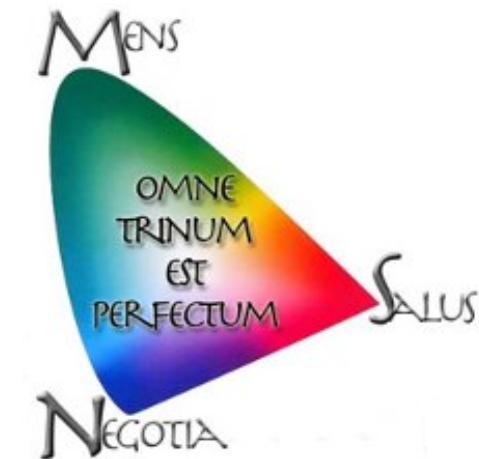


# STUDY OF FLAME RETARDANCY PROPERTIES OF NANOPARTICLE-BASED TEXTILE FABRICS

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Stresa



2010

22nd IFATCC INTERNATIONAL CONGRESS  
May 7, 2010 – Stresa (Italy)

# OUTLINE

- TOP-DOWN approach
  - Simply immersion (S\_I)
  - Plasma surface modification (PT)
  - Results
- BOTTOM-UP approach
  - Sol-gel process
  - Conclusions



# **TOP-DOWN Approach**

## NANOPARTICLES & TEXTILE FABRICS USED

### Textile fabrics:

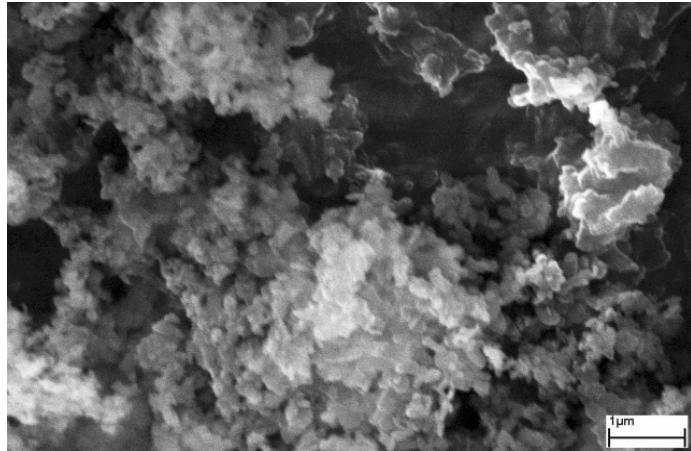
- 100% polyester (PET)
- 100% cotton (COT)
- 65% polyester + 35% cotton (PET-COT)

### Nanoparticles:

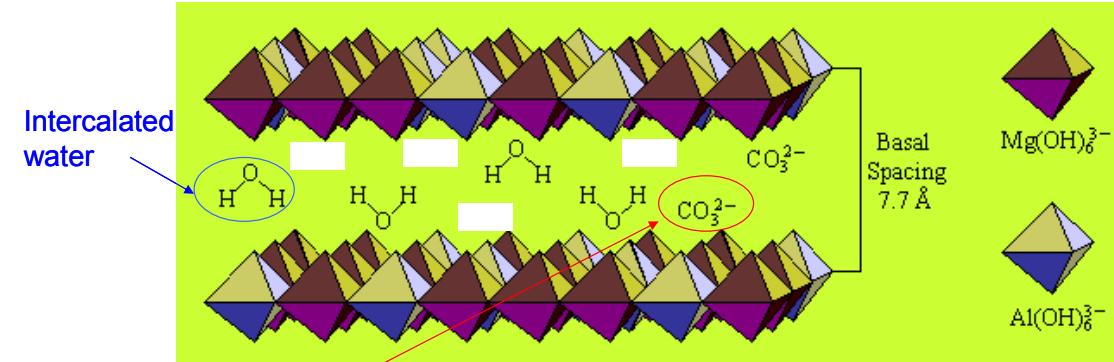
- Hydrotalcite (Pural MG63HT – Sasol)
- $\text{SiO}_2$  (Sidistar T120 – Elkem AS)



# CHARACTERIZATION HYDROTALCITE

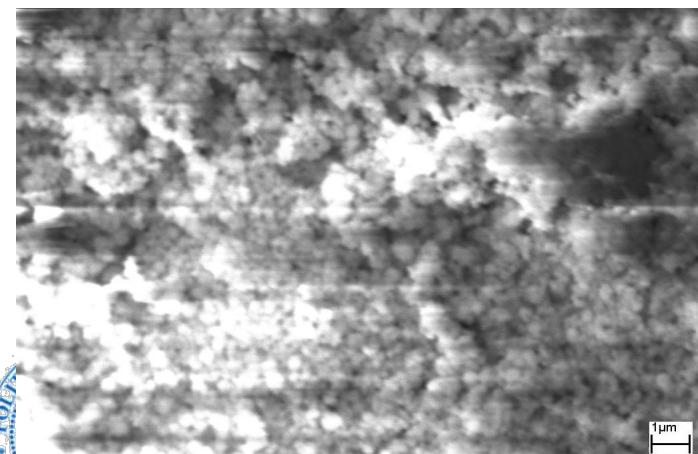


Hydroxides Layers: **positive charge**

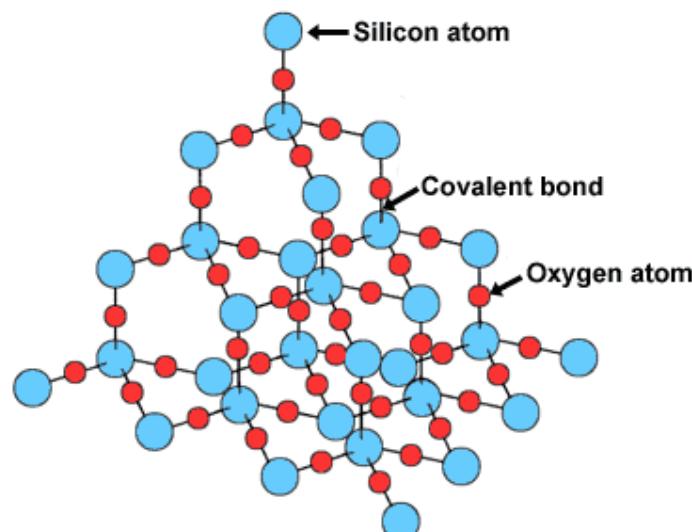


Interstitial **anions** to compensate positive layers

$\text{SiO}_2$



Nano-size spherical amorphous silicon dioxide

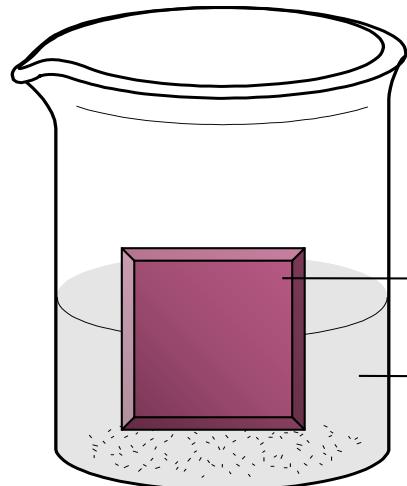


# Simply immersion

## EXPERIMENTAL SECTION

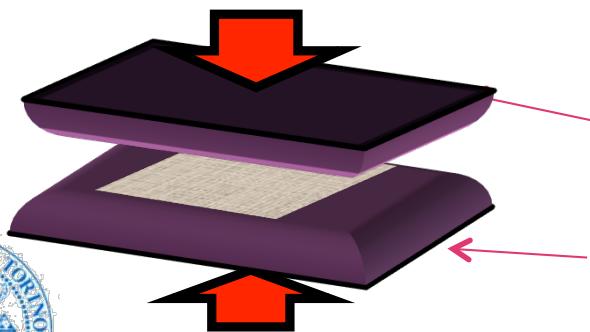
### PREPARATION OF NP-BASED TEXTILE FABRICS

- 1° step: immersion of textile into NP suspension



*Immersion time= 30 or 60 min*

- NP fixation by thermal treatment



Hot disks

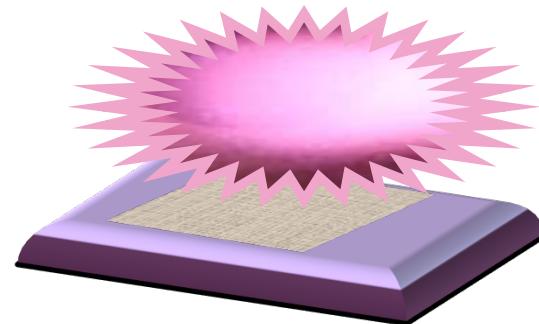
*@ T= 200°C, P= 2.5Ton  
for 10min*



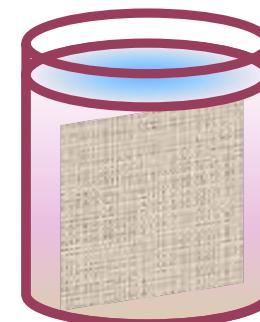
# EXPERIMENTAL SECTION

## PREPARATION OF NP-BASED TEXTILE FABRICS

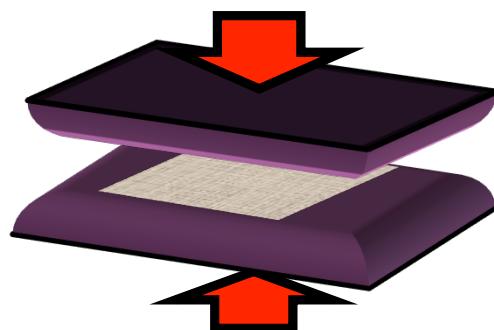
- Plasma etching of the surface ( $O_2$ )



- Immersion for 60min in a NP suspension

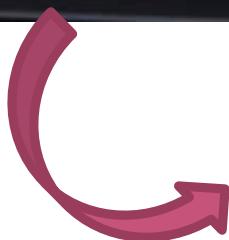


- NP fixation by thermal treatment  
@  $T=200^\circ C$ ,  $P=2.5\text{Ton}$  for 10min



# Plasma surface modifications: ETCHING APPARATUS AND CONDITIONS

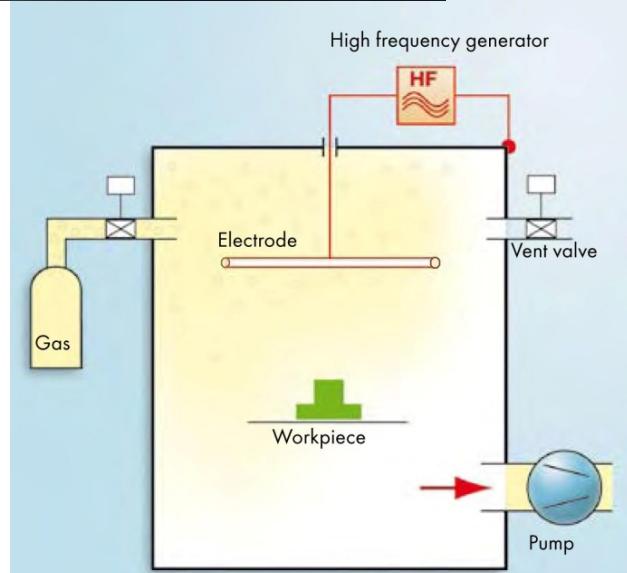
Cold plasma RF 40 kHz



Sample for etching process



Textile  
fabric  
 $20 \times 10$  cm



Etching conditions:  
Gas: O<sub>2</sub>  
Pressure:  $5 \times 10^{-1}$  mbar  
Power: 200 W  
Time: 5 min



# CONE CALORIMETRY (ISO 5660)

## THE PRINCIPLE OF OXYGEN CONSUMPTION CALORIMETRY

HRR, *Heat Release Rate*

THR, *Total Heat Release*

TTI, *Time To Ignition*

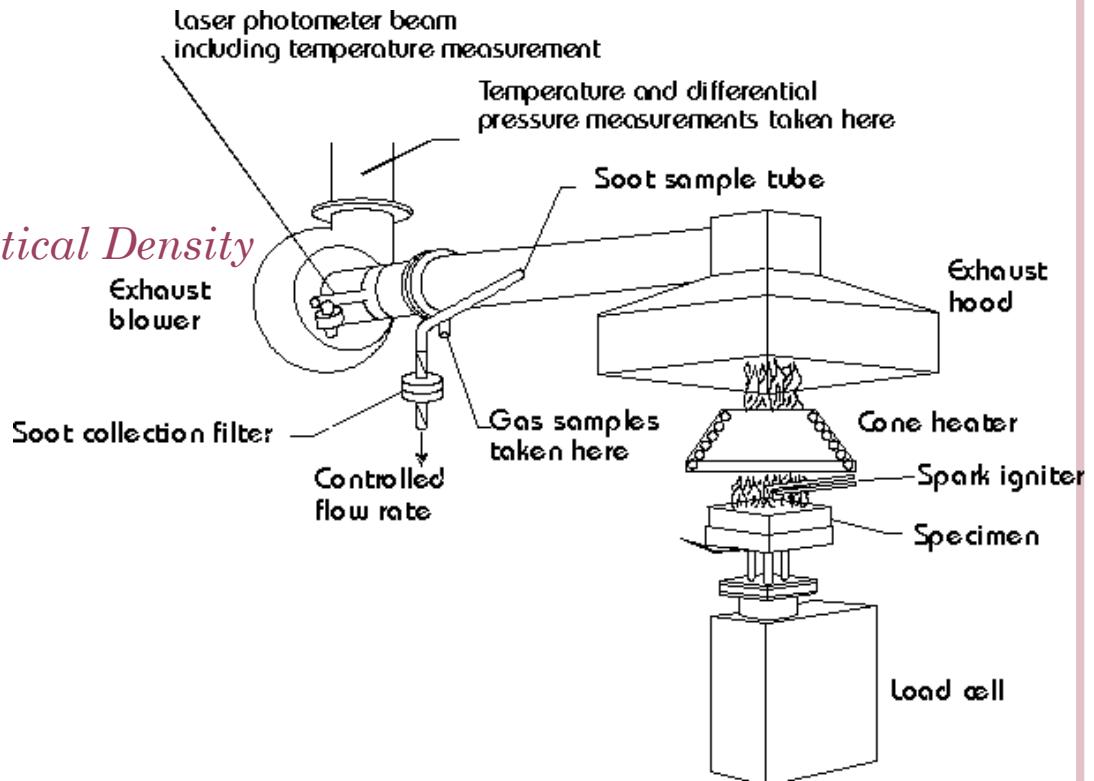
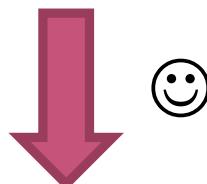
TSR, *Total Smoke Release and OD, Optical Density*

Mass or residue

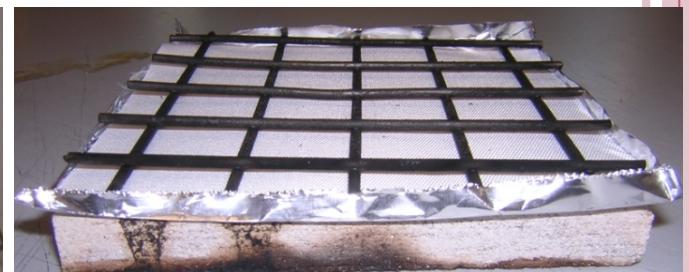
CO<sub>2</sub> and CO amount

Fire Performance Index (FPI)

$$FPI = \frac{pkHRR}{TTI}$$



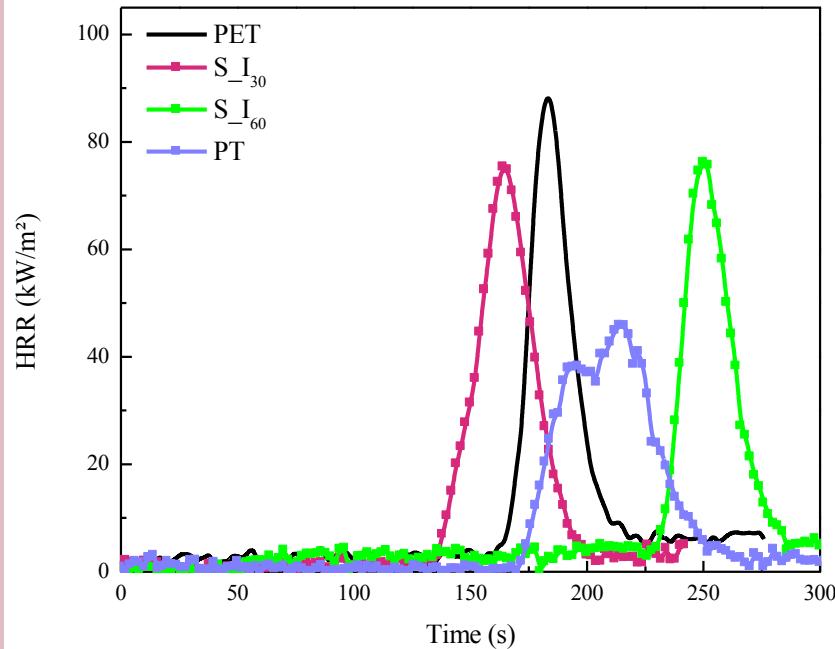
Specimen size: 100x100x0.5 mm  
Heat Flux: 35 kW/m<sup>2</sup>



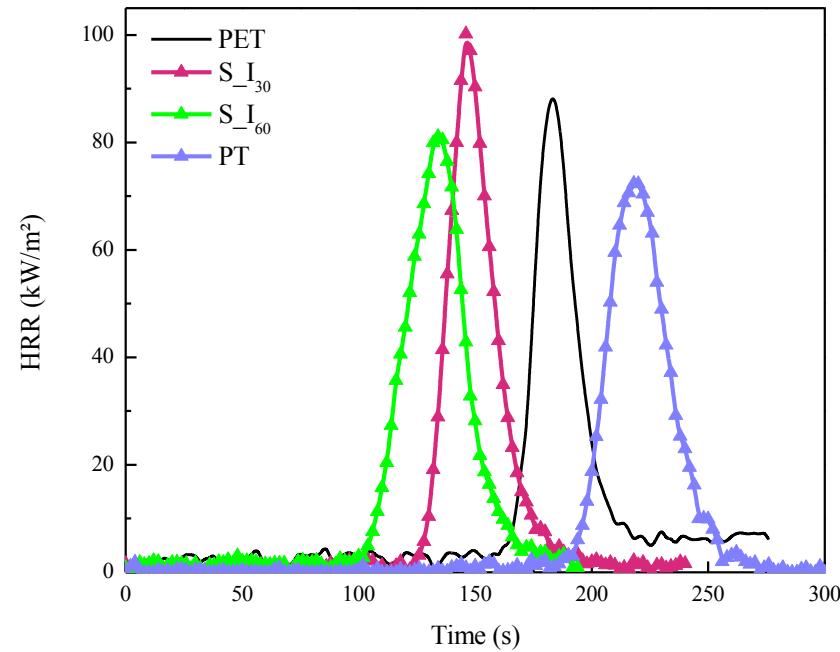
# RESULTS

## CONE CALORIMETRY

Hydrotalcite loaded textiles



SiO<sub>2</sub> loaded textiles



PET  
textiles

		TTI (s)	$\Delta$ (s)	pkHRR (kW/m²)	$\Delta$ (%)	FPI
HT	PET	166	-	95	-	0,57
	S_I <sub>30</sub>	134	-32	75	-21	0,56
	S_I <sub>60</sub>	226	+60	76	-20	0,34
	PT	170	+4	45	-53	0,26
SiO <sub>2</sub>	S_I <sub>30</sub>	122	-44	100	+5	0,82
	S_I <sub>60</sub>	105	-61	81	-15	0,77
	PT	192	+26	72	-24	0,38



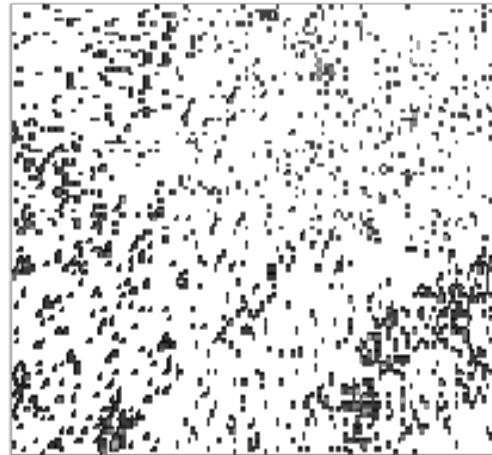
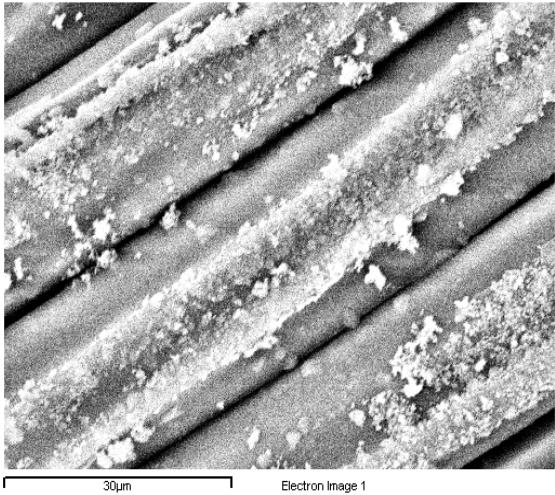
# PET textiles



## RESULTS

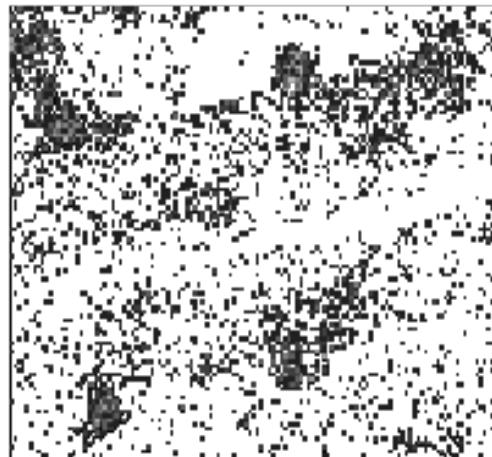
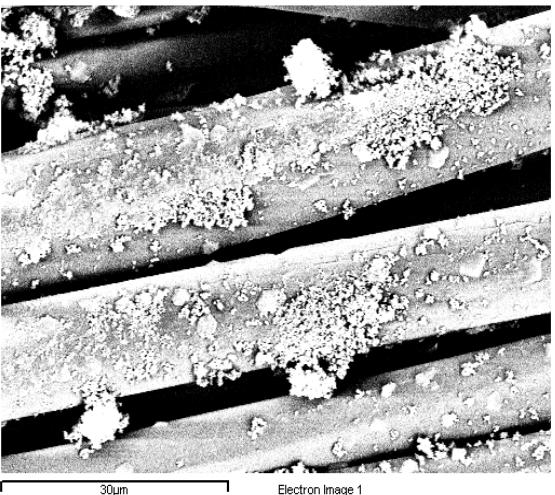
### SEM & EDS

S<sub>x</sub>I<sub>60</sub> in SiO<sub>2</sub>



Si

PT in SiO<sub>2</sub>

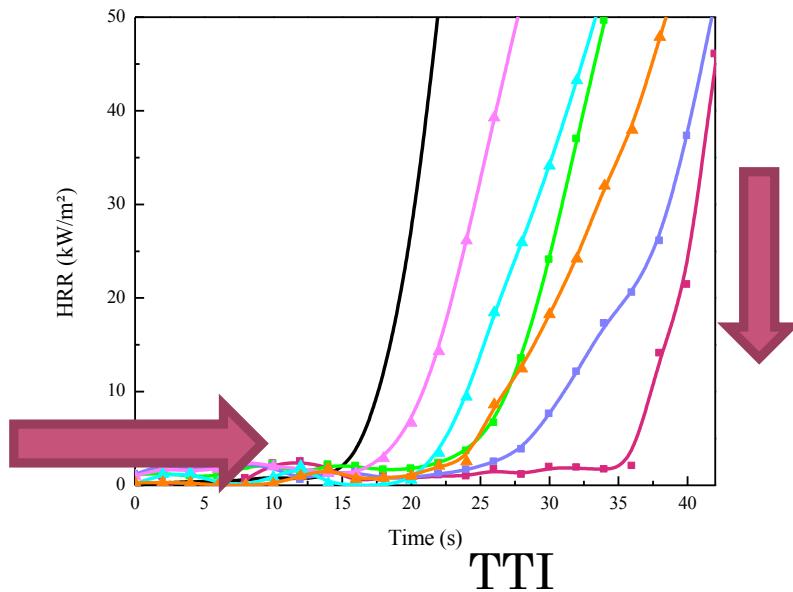
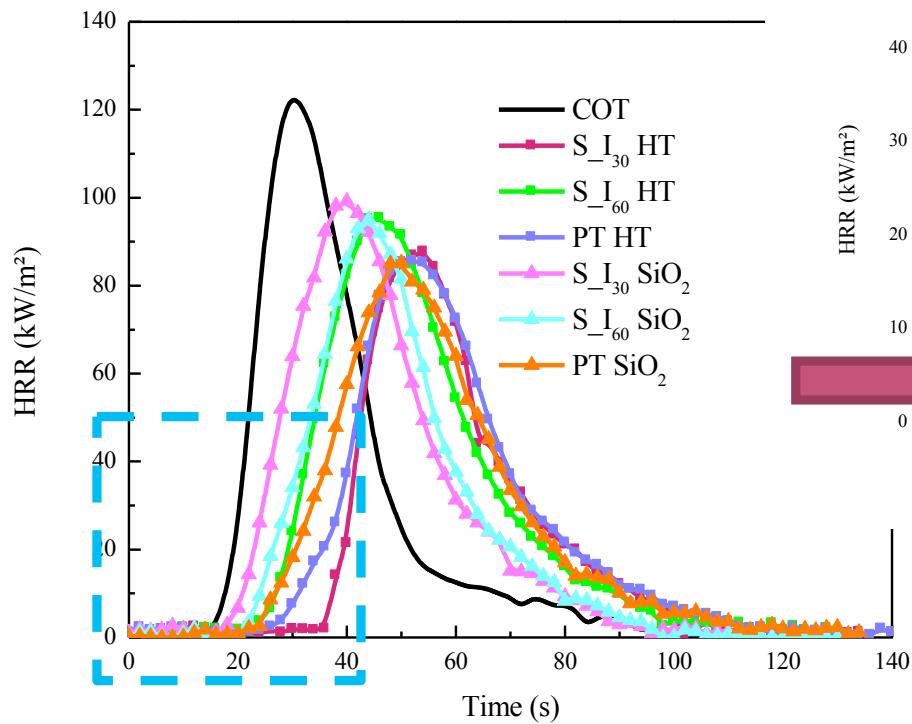


# COT textiles



# RESULTS

## CONE CALORIMETRY



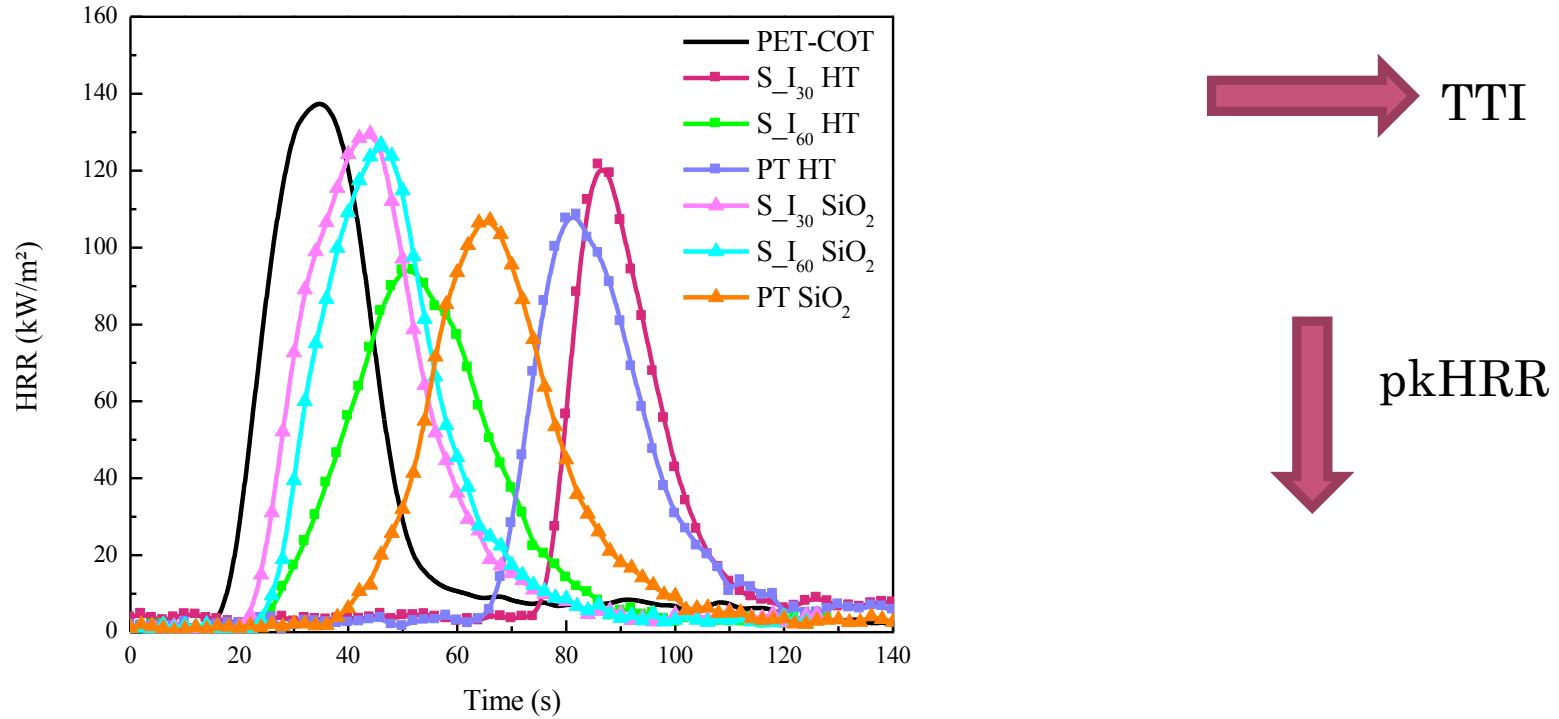
		TTI (s)	$\Delta$ (s)	pkHRR (kW/m <sup>2</sup> )	$\Delta$ (%)	FPI
	COT	14	-	124	-	8,86
HT	S_I <sub>30</sub>	36	+22	87	-30	2,42
	S_I <sub>60</sub>	22	+8	95	-23	4,32
	PT	22	+8	86	-31	3,91
SiO <sub>2</sub>	S_I <sub>30</sub>	16	+2	99	-20	6,19
	S_I <sub>60</sub>	18	+4	95	-23	5,28
	PT	20	+6	86	-31	4,30





# RESULTS

## CONE CALORIMETRY



		TTI (s)	$\Delta$ (s)	pkHRR ( $\text{kW}/\text{m}^2$ )	$\Delta$ (%)	FPI
HT	PET-COT	14	-	138	-	9,86
	S_I <sub>30</sub>	74	+60	121	-12	1,64
	S_I <sub>60</sub>	20	+6	94	-32	4,70
	PT	64	+50	108	-22	1,69
$\text{SiO}_2$	S_I <sub>30</sub>	20	+6	130	-6	6,50
	S_I <sub>60</sub>	20	+6	127	-8	6,35
PT		36	+22	107	-22	2,97

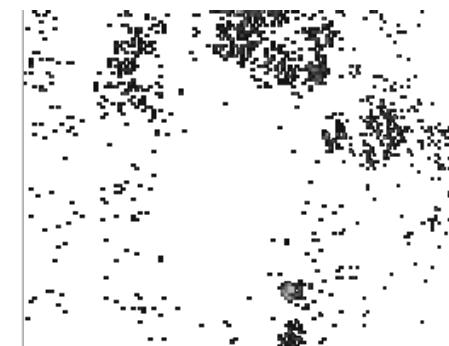
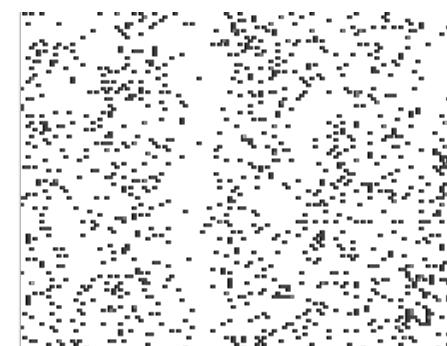
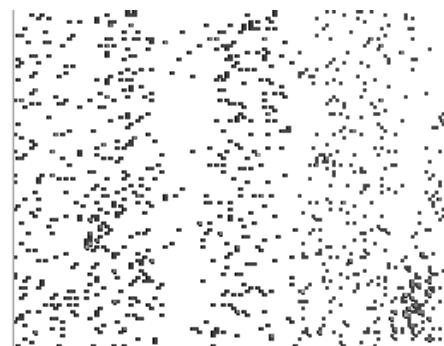
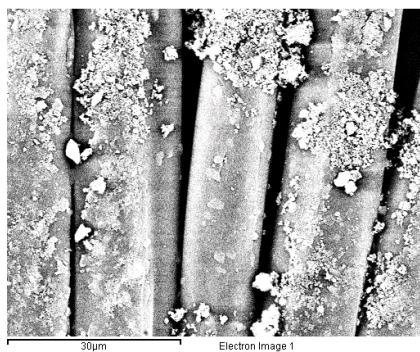


# Synergism between HT and SiO<sub>2</sub>

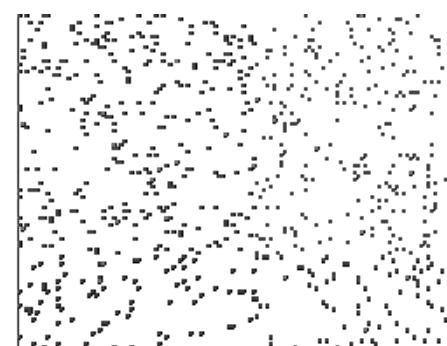
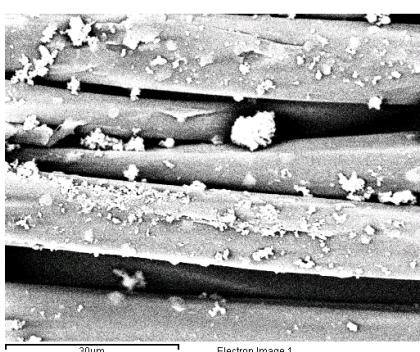
## SEM & EDS

## PET textiles

Immersed for 30min



Immersed for 60min

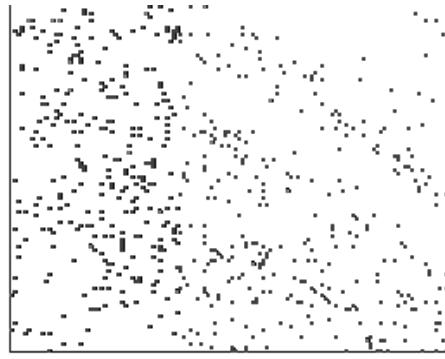
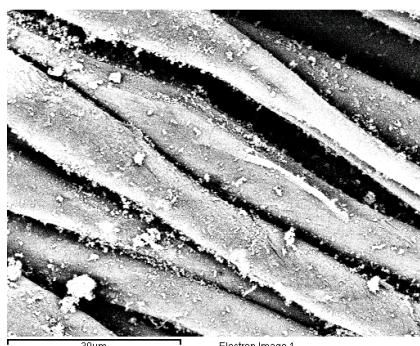


# RESULTS

## SEM & EDS

# COT textiles

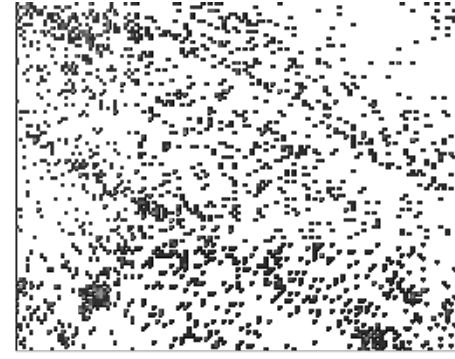
Immersed for 30min



Mg

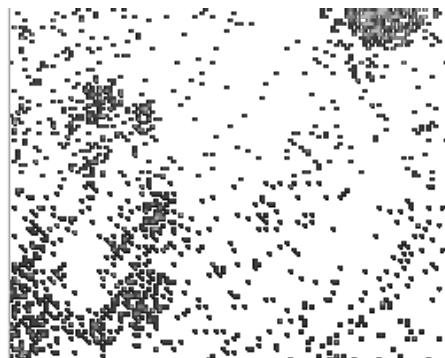
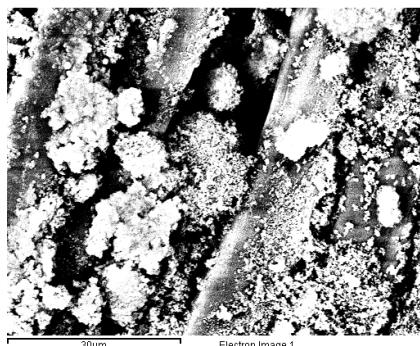


Al

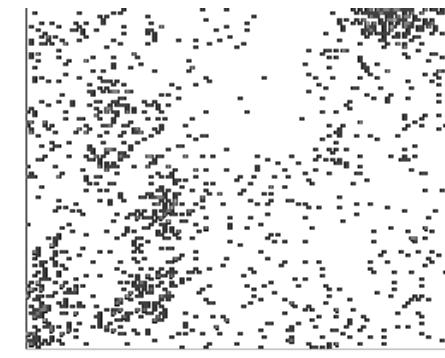


Si

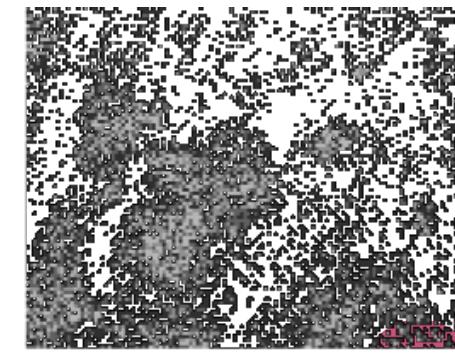
Immersed for 60min



Mg



Al

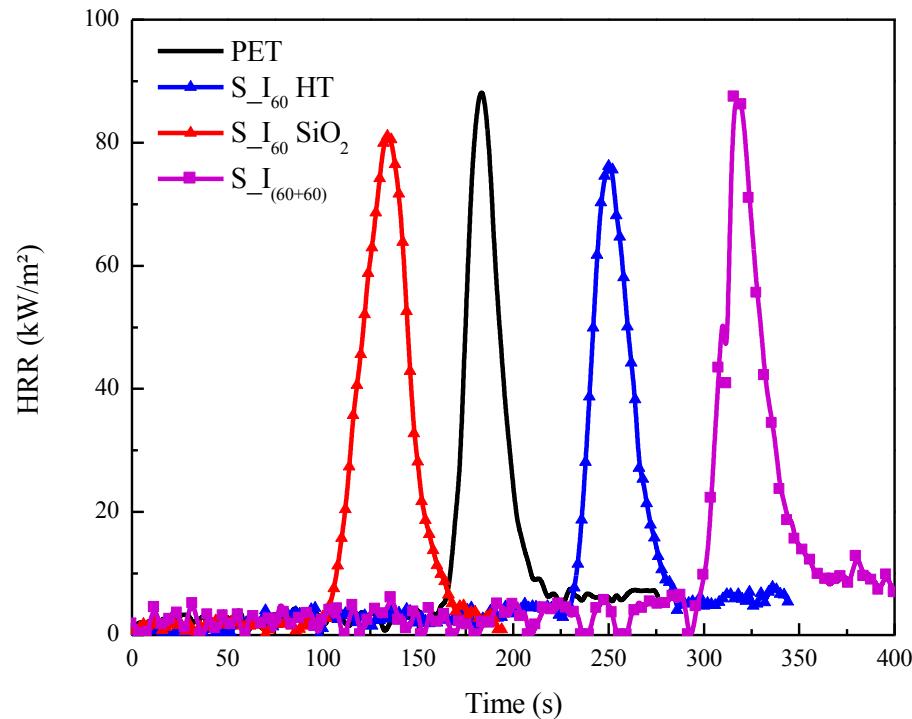


Si



# Synergism between HT and SiO<sub>2</sub>

## CONE CALORIMETRY



TTI

pkHRR

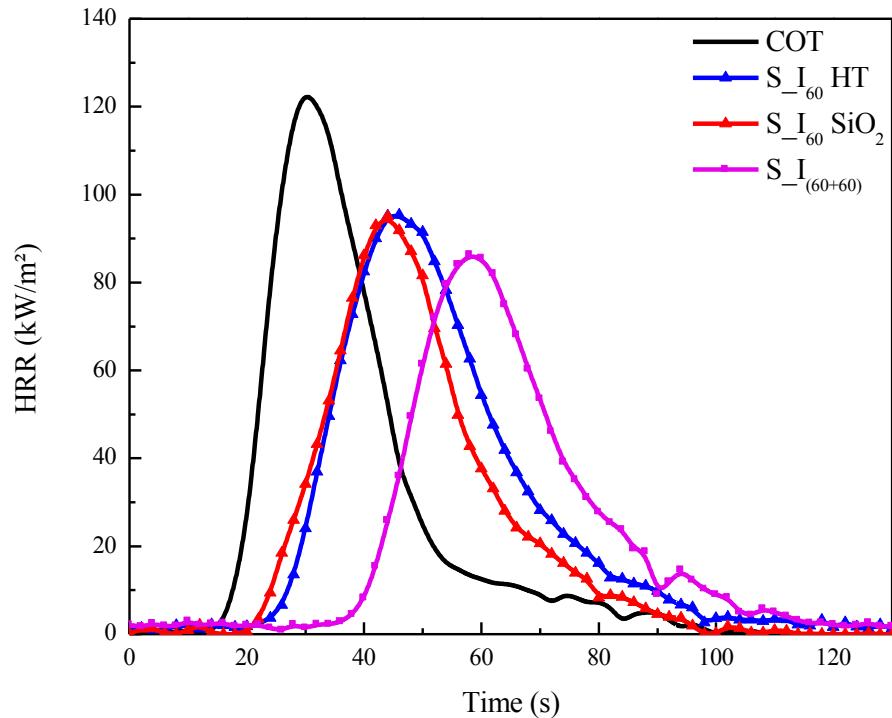
PET textiles

		TTI (s)	$\Delta$ (s)	pkHRR (kW/m <sup>2</sup> )	$\Delta$ (%)	FPI
	PET	166	-	95	-	0,57
HT	S-I <sub>60</sub>	226	+60	76	-20	0,34
SiO <sub>2</sub>	S-I <sub>60</sub>	105	-61	81	-15	0,77
	S-I <sub>(60+60)</sub>	294	+128	87	-8	0,30



# Synergism between HT and SiO<sub>2</sub>

## CONE CALORIMETRY



→ TTI

↓ pkHRR

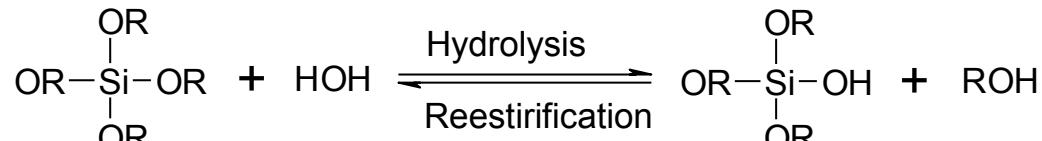
COT textiles

		TTI (s)	$\Delta$ (s)	pkHRR ( $\text{kW}/\text{m}^2$ )	$\Delta$ (%)	FPI
	COT	14	-	124	-	8,86
HT	S-I <sub>60</sub>	22	+8	95	-23	4,32
SiO <sub>2</sub>	S-I <sub>60</sub>	18	+4	95	-23	5,28
	S-I <sub>(60+60)</sub>	34	+20	86	-31	2,53

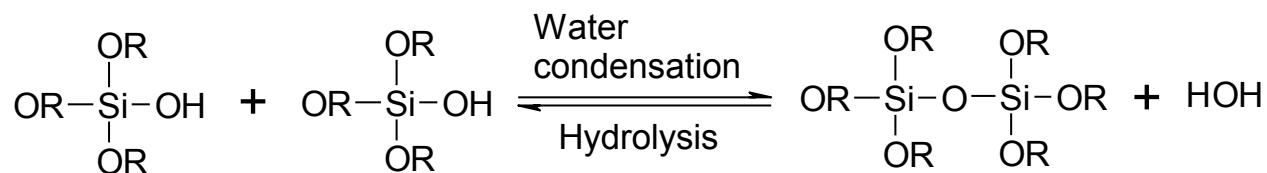


# Sol-gel Process

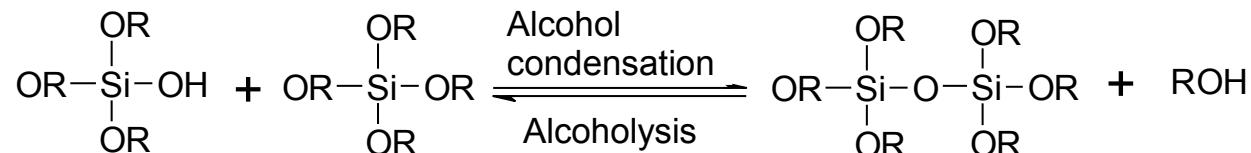
## BOTTOM-UP Approach PREPARATION OF NP-BASED TEXTILE FABRICS



(1)



(2a)



(2b)

where R= -CH<sub>2</sub>CH<sub>3</sub> Tetraethoxysilane (TEOS)

1:1

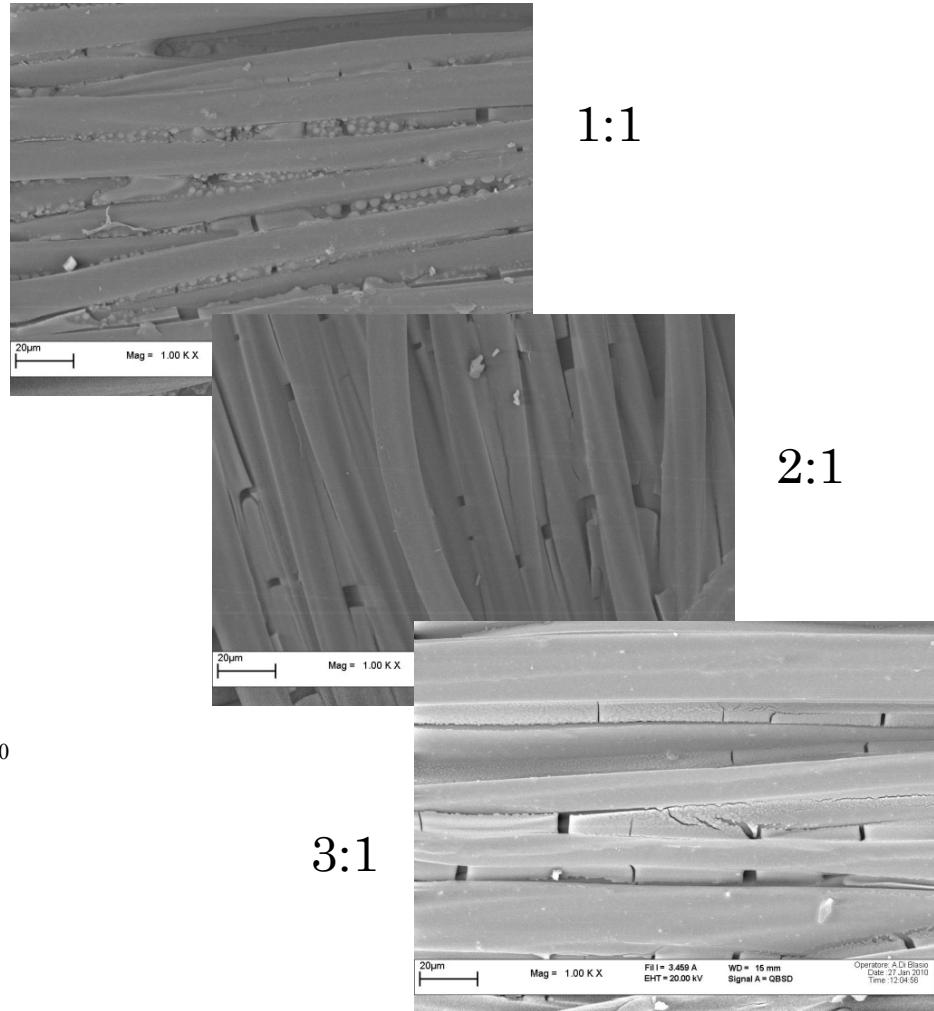
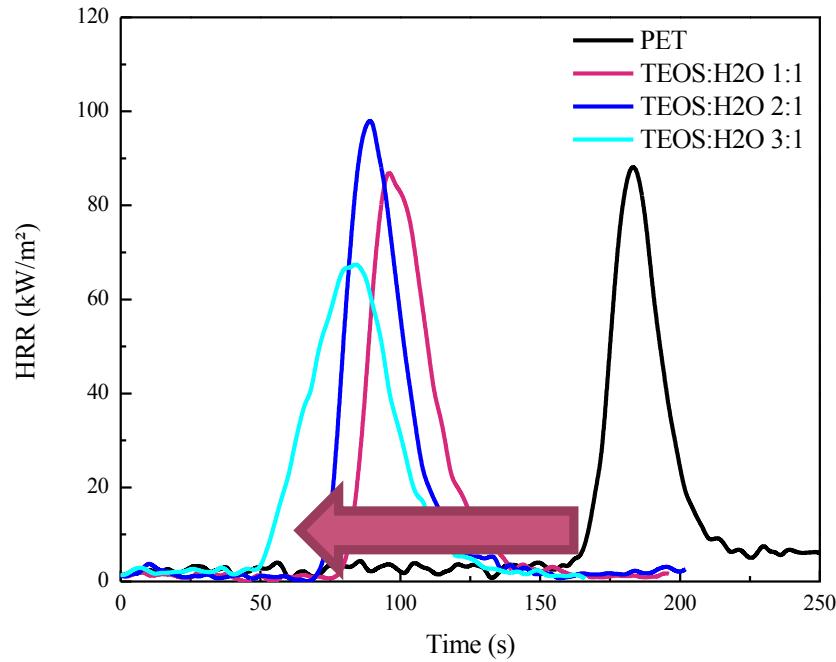
TEOS:H<sub>2</sub>O 2:1

3:1



# RESULTS

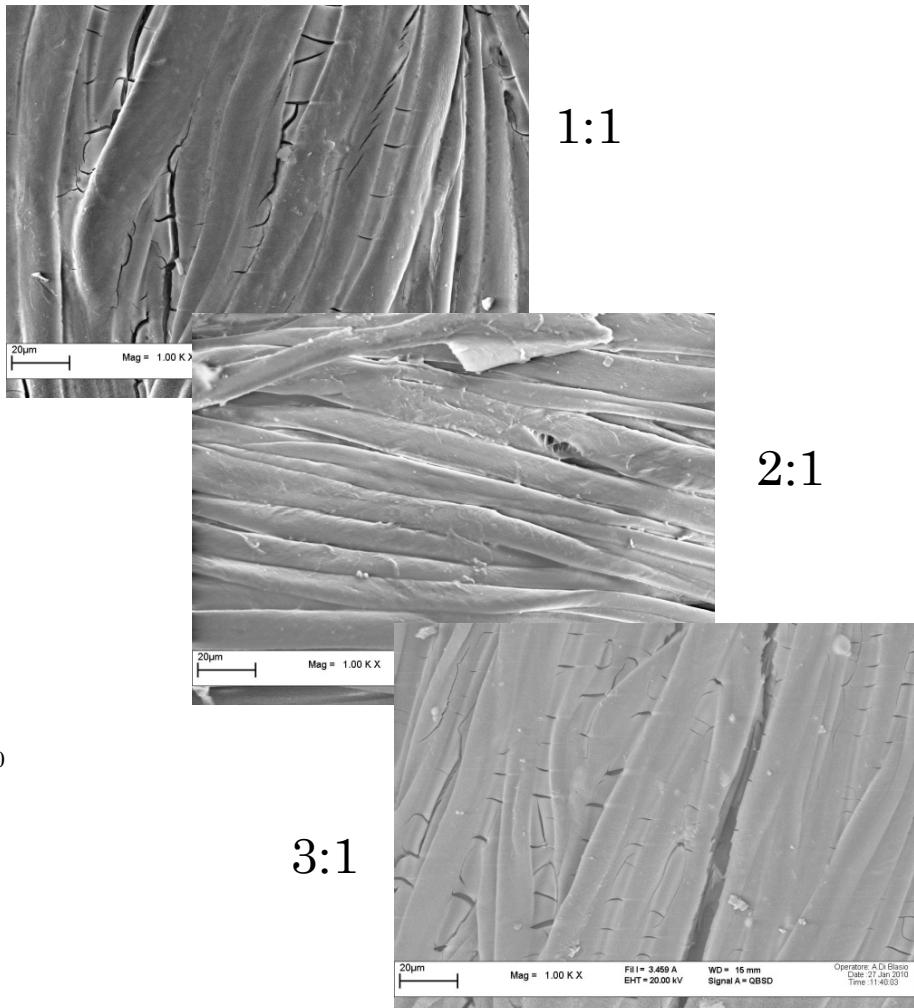
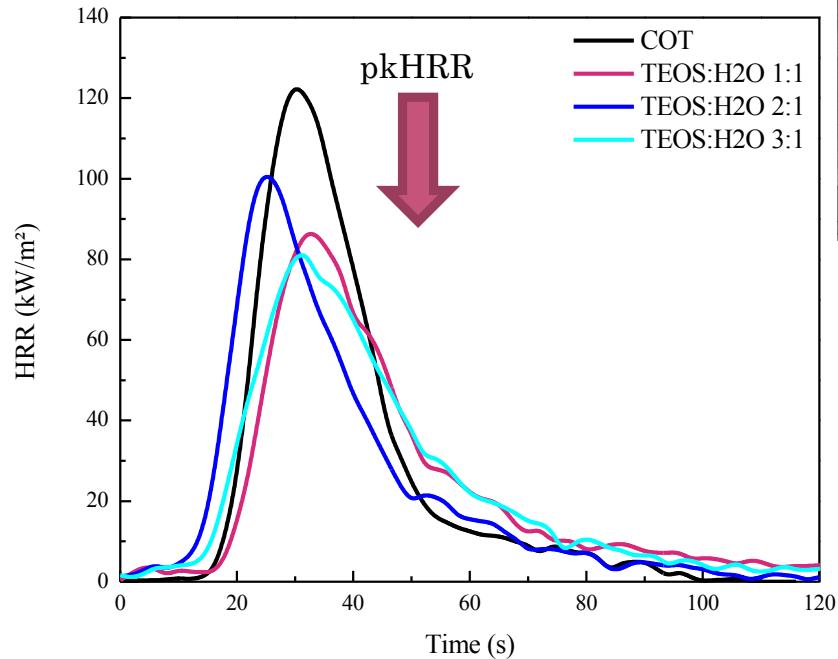
## CONE CALORIMETRY



		TTI (s)	$\Delta$ (s)	pkHRR ( $\text{kW}/\text{m}^2$ )	$\Delta$ (%)	FPI
TEOS:H <sub>2</sub> O	PET	166	-	95	-	0,57
	1:1	74	-92	88	-7	1,19
	2:1	68	-98	99	+4	1,46
	3:1	46	-120	68	-28	1,48

# RESULTS

## CONE CALORIMETRY



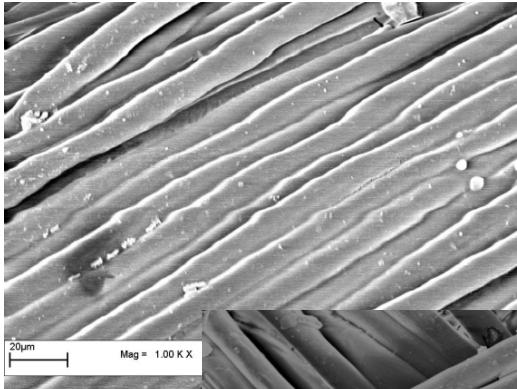
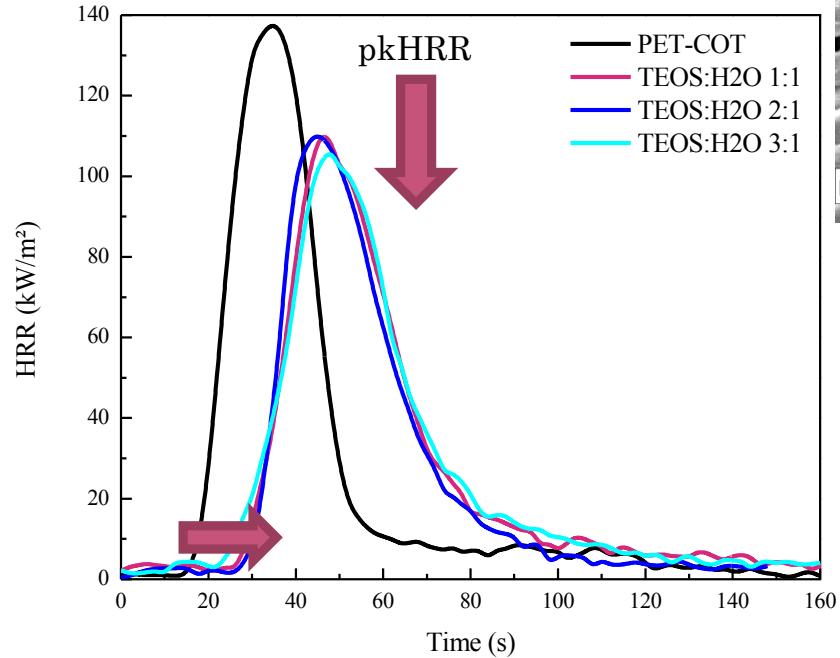
COT textiles



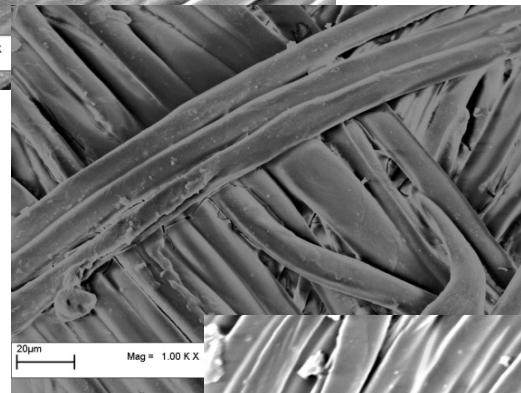
		TTI (s)	Δ (s)	pkHRR ( $\text{kW}/\text{m}^2$ )	Δ (%)	FPI
	COT	14	-	124	-	8,86
TEOS:H <sub>2</sub> O	1:1	16	+2	87	-30	5,44
	2:1	10	-4	101	-18	10,10
	3:1	12	-2	81	-35	6,75

# RESULTS

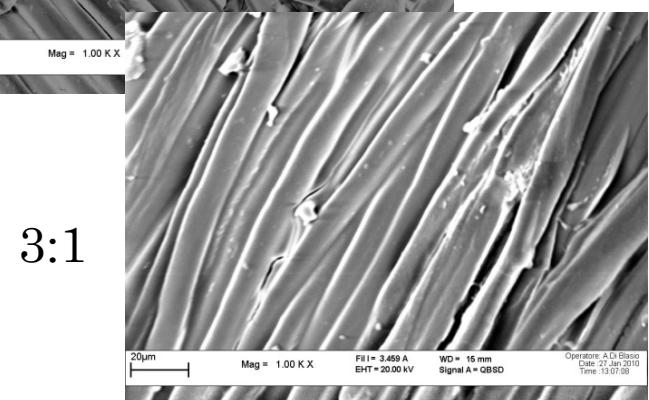
## CONE CALORIMETRY



1:1



2:1



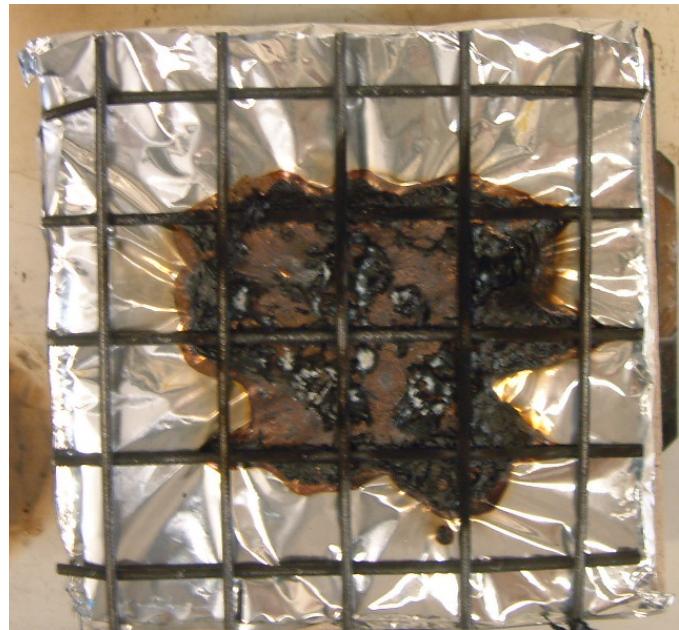
3:1

		TTI (s)	$\Delta$ (s)	pkHRR ( $\text{kW/m}^2$ )	$\Delta$ (%)	FPI
	PET-COT	14	-	138	-	9,86
TEOS:H <sub>2</sub> O	1:1	26	+12	111	-19	4,27
	2:1	26	+12	110	-20	4,23
	3:1	22	+8	106	-23	4,82



# TOP-DOWN vs BOTTOM-UP

	FPI	
	TOP-DOWN	BOTTOM-UP
PET	0.38	1.19
COT	4.30	5.44
PET-COT	2.97	4.27



# CONCLUSIONS

- Three different preparation methodologies were carried out in order to load NPs in textile fabrics.
- The use of Plasma for the surface activation enhanced the density of NPs onto the fibers.
- By the TOP-DOWN approach, the samples with the best results from the cone calorimetry point of view were:
  - COT and PET-COT blend textiles simply immersed in a HT suspension for 30min.
  - PET and COT textiles etched and further immersed in  $\text{SiO}_2$  suspension.
  - PET etched textiles in HT and  $\text{SiO}_2$  suspensions.
  - Synergic effect with the use of both HT and  $\text{SiO}_2$  NPs.
- By the BOTTOM-UP approach, the samples with the best results from the cone calorimetry point of view were only the fabrics of COT and PET-COT blend with a molar ratio TEOS:H<sub>2</sub>O 1:1.

## Articles:

- 1) “Investigation on thermal stability and fire resistance of polyester, cotton and relative blend textile fabrics subjected to sol-gel treatments”, Jenny Alongi, Mihaela Ciobanu, Federico Carosio, Jennifer Tata, Giulio Malucelli, submitted to *Journal of Applied Polymer Science*.
- 2) “Influence of cold plasma on thermal and fire stability of PET and cotton textiles treated with nanometric silica and hydrotalcite”, Jennifer Tata, Jenny Alongi, Alberto Frache, *on preparation*.
- 3) “Optimization of the procedure to burn textile fabrics by cone calorimeter: Part I. Combustion behavior of polyester”, Jennifer Tata, Jenny Alongi, Federico Carosio, Alberto Frache, submitted to *Fire and Materials*.



# Politecnico di Torino – Alessandria Branch



Thank you