Università degli Studi di Padova – Dipartimento di Ingegneria Industriale

Corso di Laurea in Ingegneria Chimica e dei Materiali

Relazione per la prova finale «Effect of water vapour and methanol treatment on silk fibroin/TiO_2 nanocomposite film»

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SILK AND SYNTHESIS OF SF SOLUTION

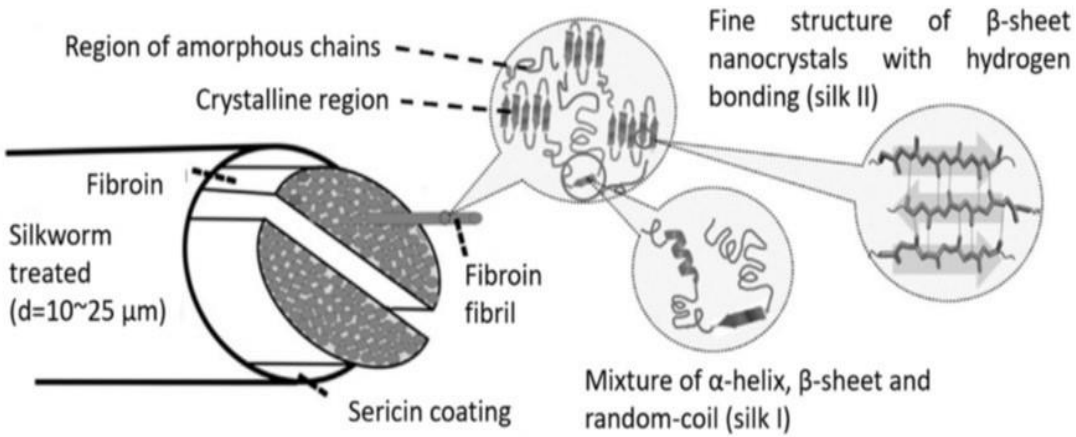
Silk

- great mechanical properties, hygroscopicity and biocompatibility

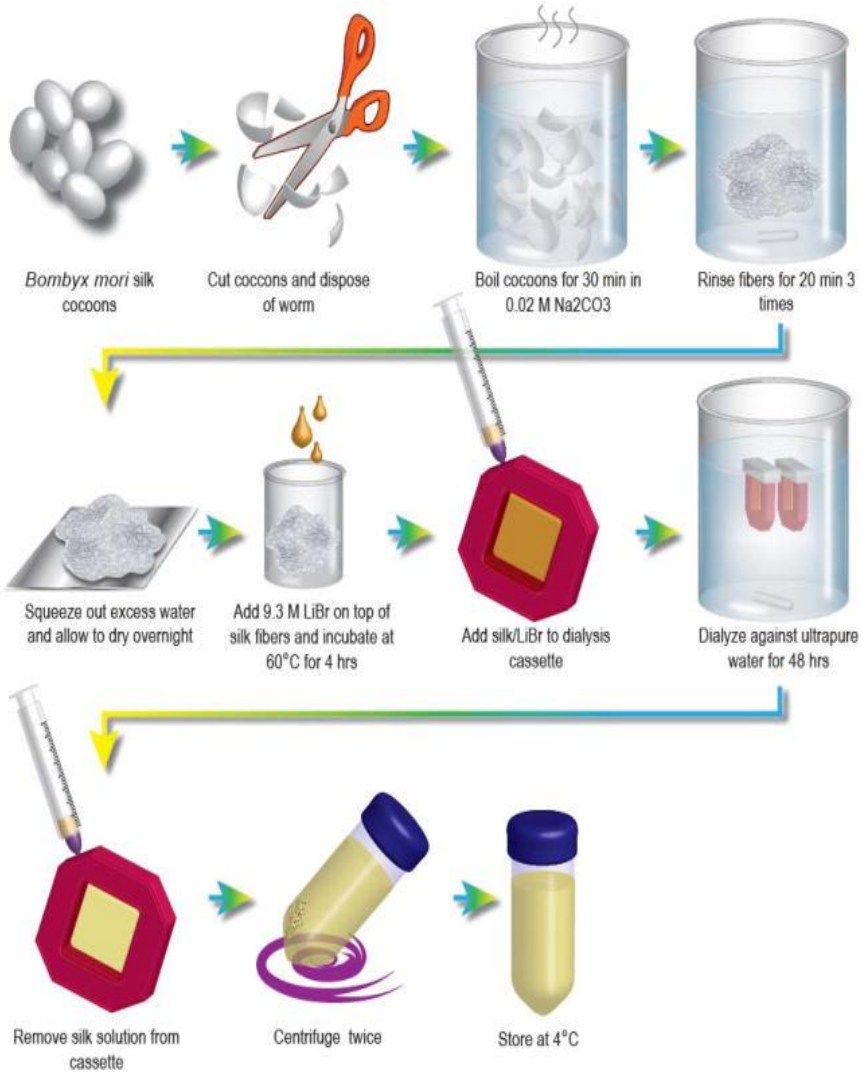
- 2 proteins: sericin and fibroin

- 4 possible configurations of fibroin: α helices, β sheets, random coils and turns

- crystalline regions (silk II and silk I) and amorphous regions are intercalated in the fibroin resulting in a combination of toughness and resistance



Synthesis of SF solution



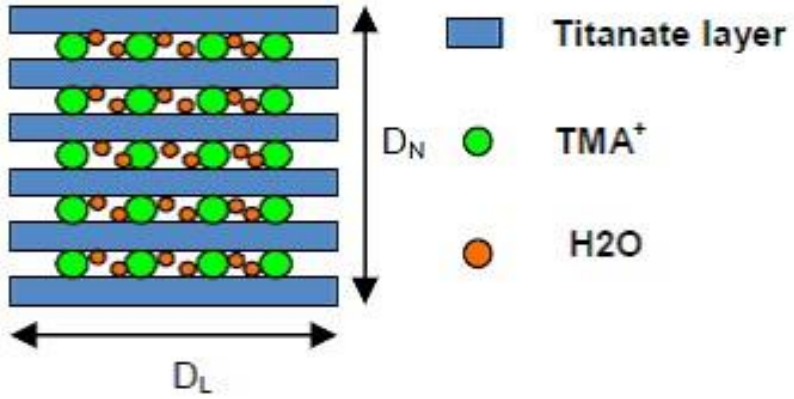
Layered titantes

- 2D layered compounds

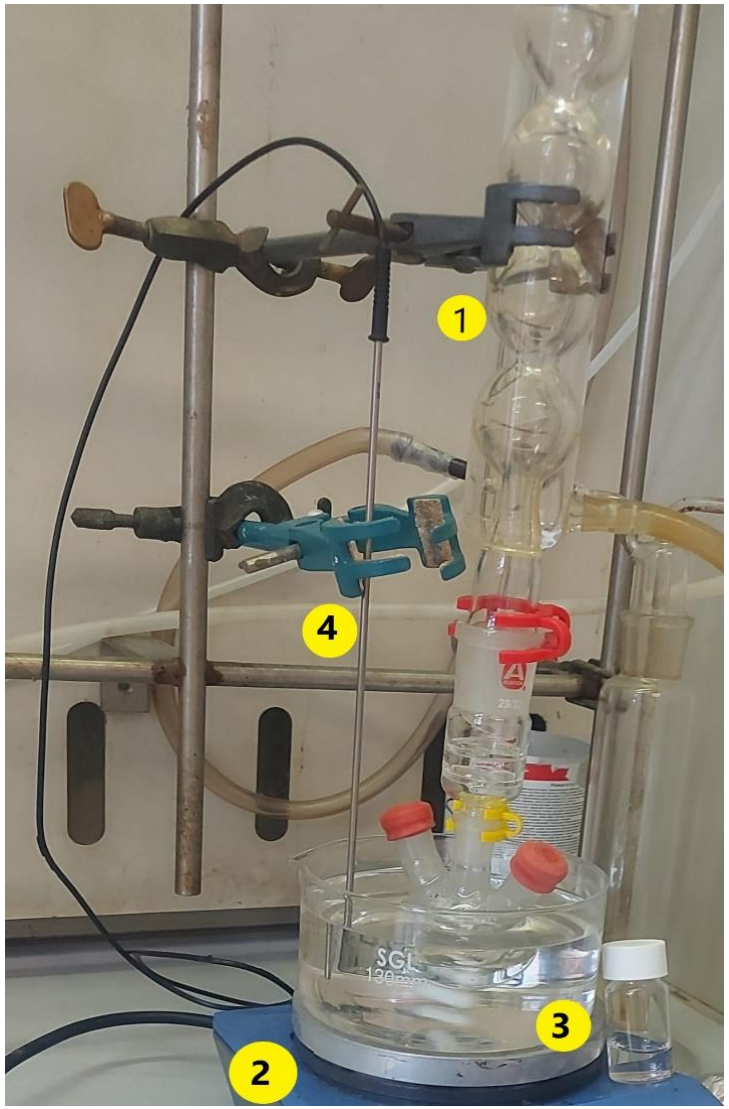
- structure of $[Ti_nO_{2n+1}]^{2-}$ nanosheets intercalated with a guest cation (TMAH)

- particular nanometric structure

- good candidates for optoelectronic applications



Synthesis of TNS solution (sol-gel method)



WHY COMBINE SILK AND TITANTES?

TNS/SF COMPOSITES

- combine the biocompatibility and mechanical properties of the fibroin with the optical properties of the layered titanates

- titanates can be processed easily => integration with silk is fairly simple

- nanometric dimension grants a good and quite uniform dispersion in the composite

PURPOSE OF THE STUDY

This study will have 2 main purposes:

- ***understand and implement*** a nanocomposite obtained by blending suitably processed silk fibroin with layered titanates

- ***investigate*** the treatment ***affects on the secondary structure*** of the compound and the interaction between the silk fibroin and the titanates

RESULTS

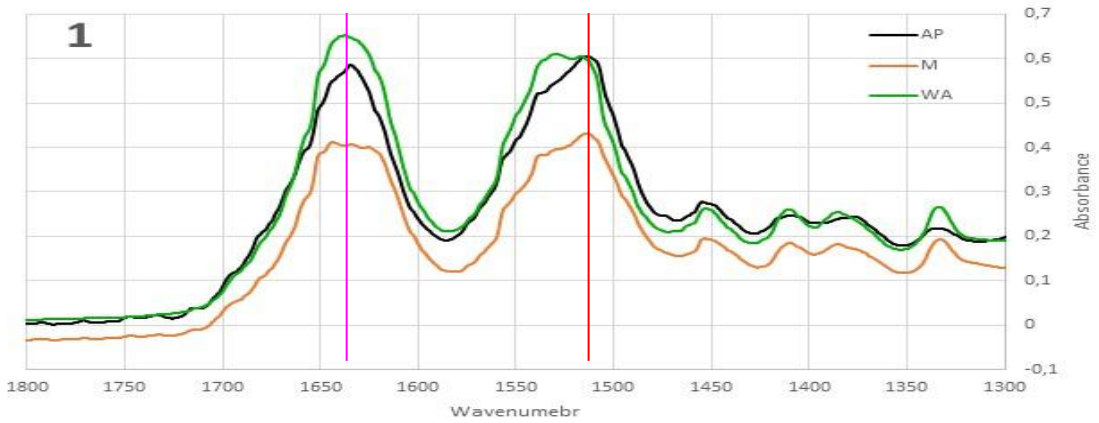
Sample composition	Total V [mL]	V of SF solution [mL]	V of TNS solution [mL]	Average thickness [μm]
0:100 TNS/SF	3	3	/	143 ± 37.96
25:75 TNS/SF	3	2.769	0.231	149 ± 28.55
50:50 TNS/SF	2.5	0.5	2.5	187 ± 7.05
75:25 TNS/SF	2.5	1.434	1.066	319 ± 67.42

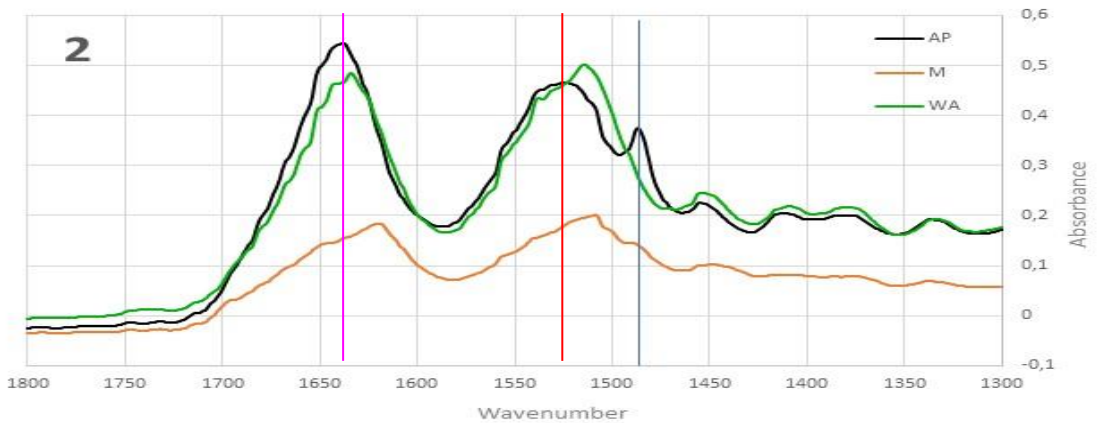
Volumes used and average samples thickness

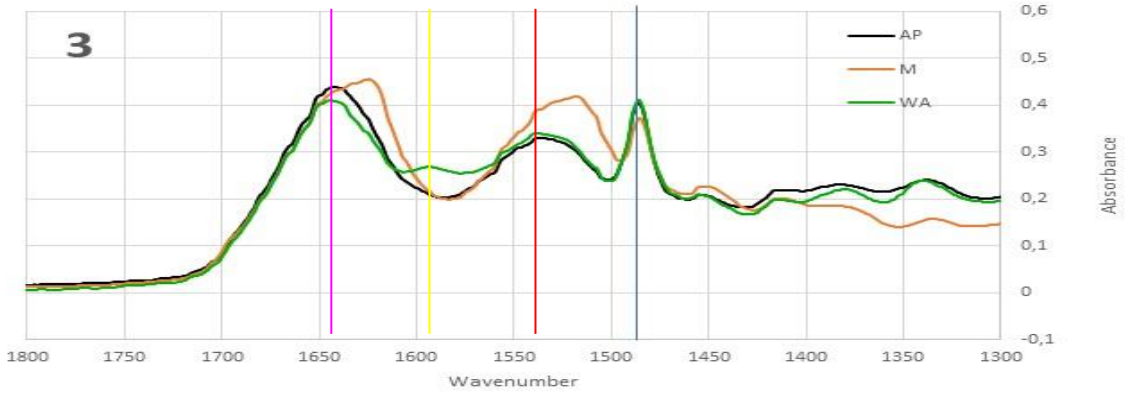
Characterisation techniques

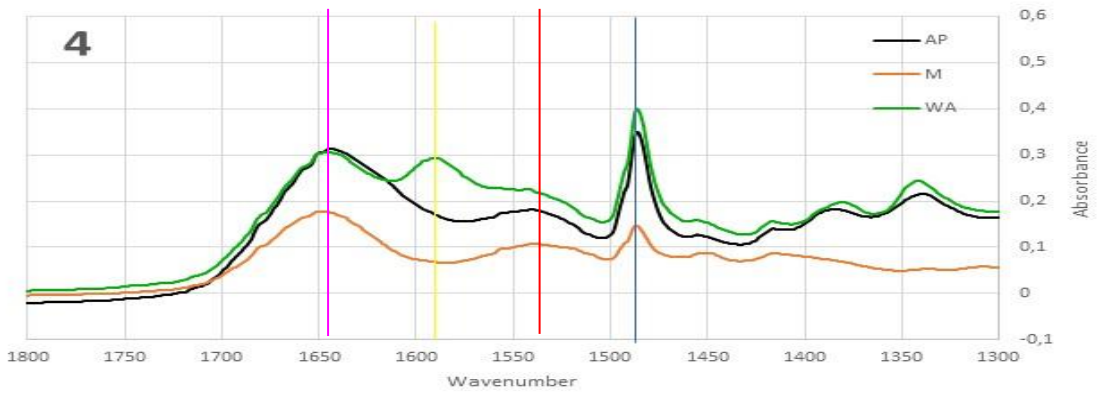
- FTIR spectroscopy
- SEM-EDX analysis

RESULTS (FTIR)









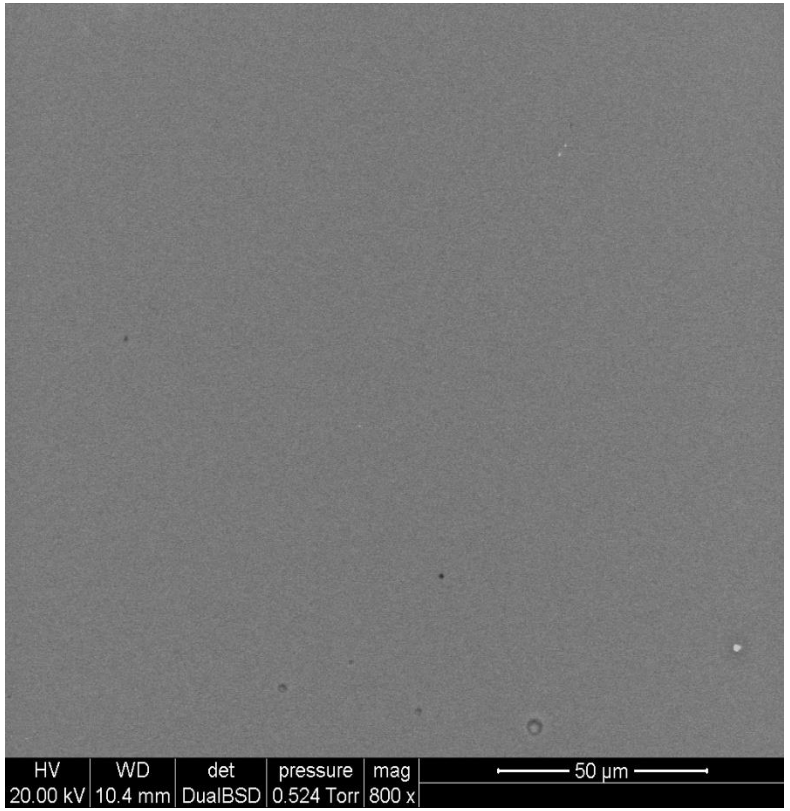
Methanol

- visible shift in both peaks
- increase in β sheets for 25:75 and 50:50 TNS/SF -> silk II
- little sifht in 75:25 TNS/SF -> silk I
- area of TMAH peak (~1488 cm^{-1}) visibly reduced in 25:75 TNS/SF sample

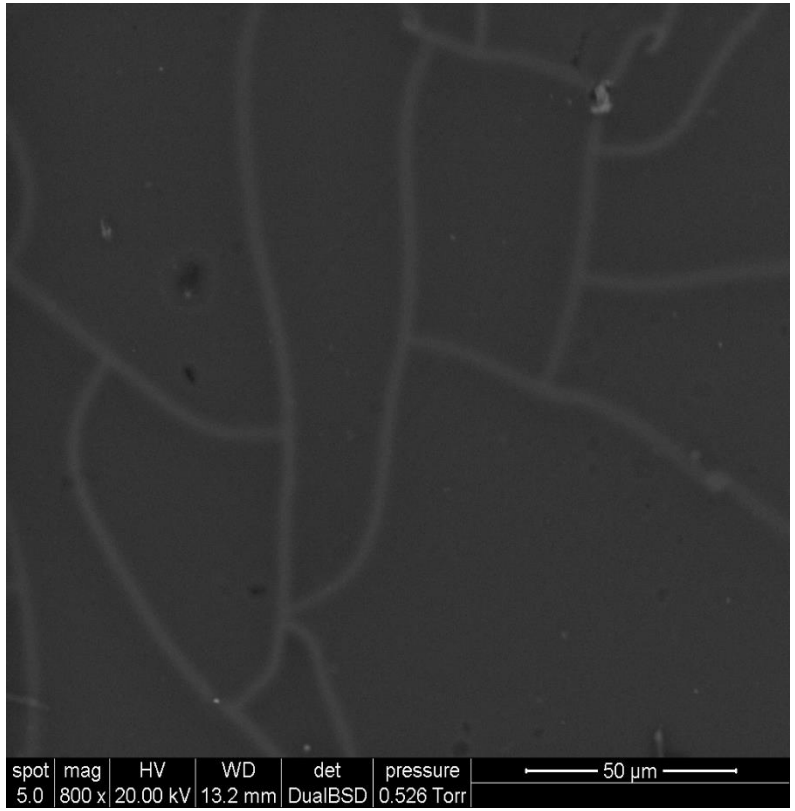
Water annealing

- less evident shift in both peaks
- similar secondary structure to the reference samples
- peak at ~1590 cm^{-1} for 50:50 and 75:25 TNS/SF samples
- similar effect in reduction of TMAH for low concentration of TNS but a bit less effective for higher concentrations

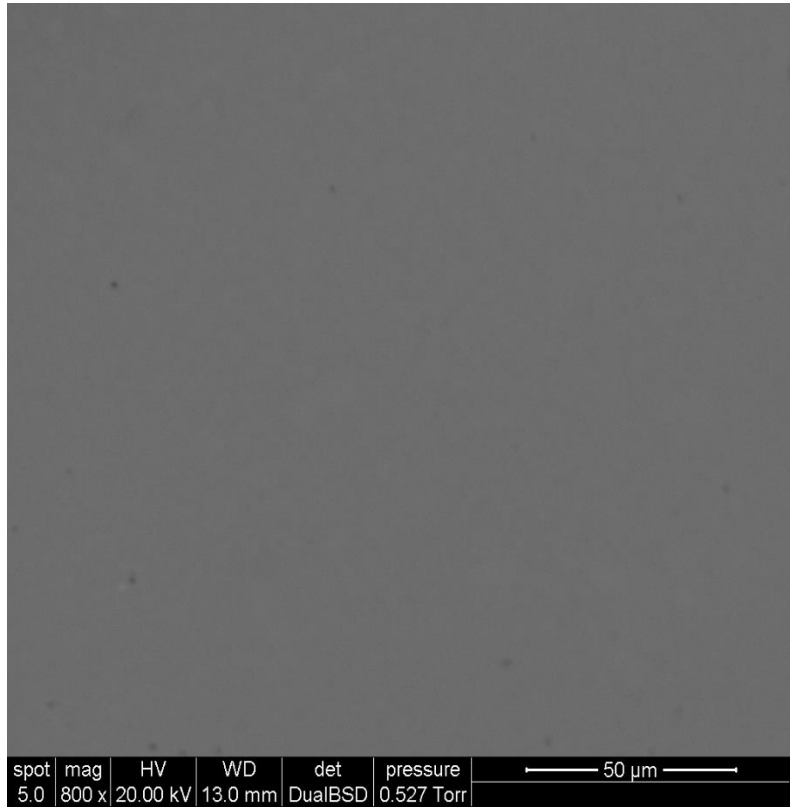
RESULTS (SEM)



50:50 TNS/SF, reference



25:75 TNS/SF, methanol



75:25 TNS/SF, water annealing

Methanol

- cracks due to dehydration
- rough morphology
- fragile and brittle samples after treatment

Water annealing

- smooth and flat surface
- similar morphology to the reference sample
- jelly-like texture post treatment (no dehydration)

CONCLUSIONS

For methanol treatment:

- decrease in TMAH content, giving better results with samples at higher titanates concentration

- slight changes in the secondary structure => formation of β sheets

- samples with higher concentrations may have impediments in reorganising => silk I

- samples are particularly fragile and brittle

For water vapour treatment:

- decrease in TMAH content but not as much as methanol

- not particularly effective with samples with a high titanates concentration

- slight changes the position of the FTIR peaks but overall no big changes in the secondary structure

- samples are almost jelly-like

Thank you for your kind attention