

# Surface Analyses of Pulp and Paper

## INTRODUCTION

Paper making and printing processes involve many surface chemical interactions. Stock preparation, coating, calendering and printing processes are steps where surface properties play an important role for the final product. Interactions between fibres, polymers, minerals and dyes are of fundamental importance in papermaking.

Surface chemical composition, interactions and physical and chemical structure may be analyzed with surface sensitive equipments. Both surfaces and sample cross sections may be studied. Information about topography can be achieved with imaging. Homogeneity and distribution of elements and ions can be studied by using mapping techniques.

## ANALYTICAL TOOLS AND EXAMPLES

**TOF - SIMS** - Time-of-flight secondary ion mass spectrometry

**ESCA (XPS)** - X-ray photoelectron spectroscopy

**SEM + EDX** – Scanning Electron Microscope + X-Ray Analyzer

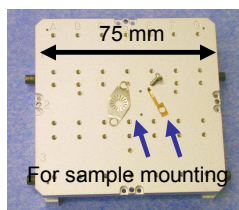
**FTIR + ATR, DRIFT and RAIR**

**Contact angle** apparatus

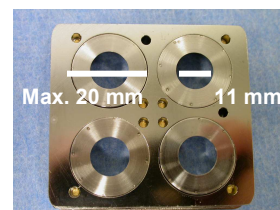
Gloss and Color measurements

Top Analytica's equipments are listed in this section. Especially SEM-EDX, ESCA and TOF-SIMS are used for paper analyses.

ESCA  
sample  
holder



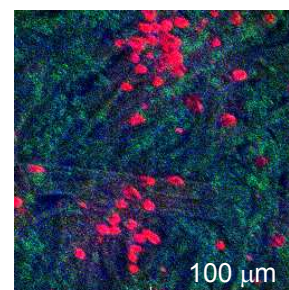
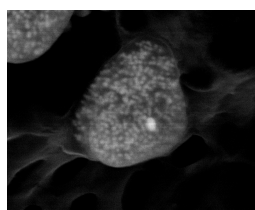
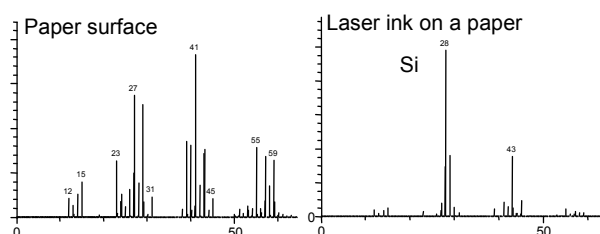
TOF-  
SIMS  
sample  
holder



### TOF-SIMS

- *analyzing depth < 2 nm*
- *qualitative elemental and molecular information*
- *organic and inorganic ions*
- *spectral information*
- *mapping (area up to 3 mm x 3 mm)*
- *surface analyses*
- *cross sections, layered structures*
- *detection limit ppm range (at best)*
- *identification of large ( $M_w \sim 1000$  amu) molecules.*

The outermost surface composition can be analyzed with TOF-SIMS technique. Below is shown TOF-SIMS spectra of a paper surface and laser ink and image of black laser ink on a paper (red = Si, blue = total ion, and green =  $C_3H_5$ ). Pigment particles (see SEM image below) are Fe-ox according to EDX-analysis. Particles are coated / covered with silicon according to TOF-SIMS.

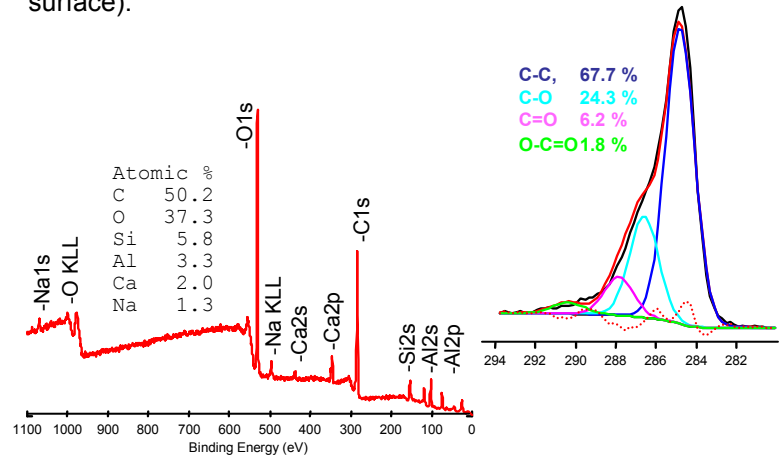


## ESCA (XPS)

- analyzing depth 2 – 5 nm
- surface chemical composition
- semi-quantitative information
- chemical binding states
- detection limit 0,1-1 atomic-%
- depth profiling
- minimum lateral resolution 10 micrometer



ESCA spectrum and binding states of carbon (C1s) of a paper surface are shown below. Surface chemical composition (atomic %) can be analyzed with ESCA technique. Carbon – oxygen binding states may be related to the reactivity of surface (type and amount of functional groups present on surface).



## HRSEM + EDX

- surface imaging (resolution 2 nm)
- surface topography
- qualitative and quantitative elemental analyses
- analyzing depth ~ micrometer
- cross sections
- mapping (resolution ~ micrometer)
- detection limit 0.1-0.5 wt-%
- from stereographic images possibility to determine surface roughness

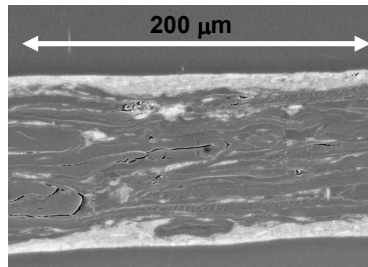
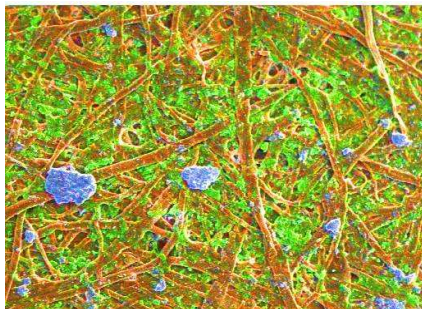
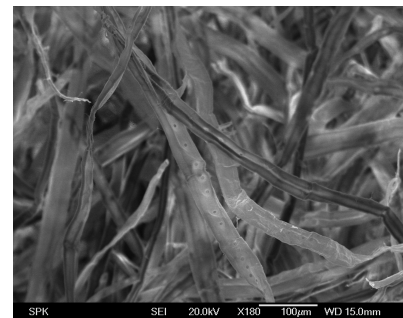
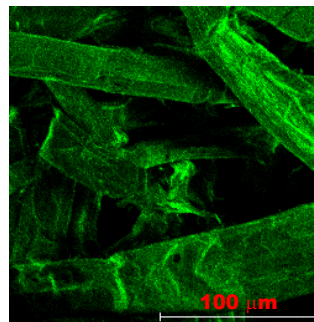


Image of a cross section of a paper. Layered structures of coated paper can be analyzed with SEM-EDS technique.



SEM image of a paper surface and (EDS) mapping of  $\text{CaCO}_3$  (blue), kaoline (green) and C, O (red).



TOF-SIMS image (total ion mapping) is shown on the left and SEM image (x180) of Pine Unbleached Kraft Pulp on the right. Morphology of fiber surfaces can be studied by SEM and/or TOF-SIMS imaging. The images of pulps have been done by Dr. Pedro Fardim at Åbo Akademi University.