Course: Puu-0.3130 Instrumental Analysis in Surface, Polymer and Nanoscience

Written examination duration: 3 hours

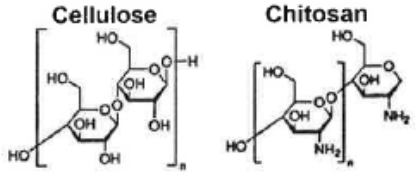
Instructions: Answer any 5 of the below questions. The approximate length of answer expected is also included. One can choose to answer more than 5 questions in which case 5 of the highest scoring answers shall be considered for grading purposes.

Question 1. (5 points 3+2)

a. What are the main reasons for a surface to develop electrical charge? How will you determine the surface charge of a surface? How will you determine zeta potential of a surface (mention any one technique) ?(3 points 1-2 pages)
b. The zeta potential of unmodified paper is –25 mV at pH 7. At pH 2 and pH 10, the zeta potential of paper was measured to be –5 mV and –30 mV, respectively. Relative to unmodified paper explain qualitatively (nature of change) and quantitatively (extent of change) the effect of two different pH conditions on surface charge of paper. (2 points ½ page)

Question 2. (5 points 3+2)

a. Explain photoelectric effect using Koopmans theorem with respect to x-rays? Explain based on binding energies how elemental composition of surface is determined. What information does one derive from high resolution XPS spectrum? (3 points 1-2 pages)
b. Consider the chemical structures of Cellulose and Chitosan given below. How will they differ when analysed using survey (wide scan) XPS spectra? How will they differ in high resolution C1s XPS spectra? (2 points ½-1 page)



Question 3. (5 points 1+2+2)

a. Explain why Infrared radiation with specific frequency is absorbed by a vibrational mode of a bond with non-zero dipole moment? (1 point ½ page)
b. In short explain sample preparation procedures involved in Infrared spectroscopy. If your sample has water in it, which of the two (Raman or IR) vibrational spectroscopies would you choose and why? (2 points 1-2 pages)
c. Consider the bonds C-C, C-N and C-F. Given the order of dipole moments C-C < C-N < C-F, what is the expected order with respect to strength of Raman peak (strong, medium and weak) and IR peak (strong, medium and weak)? In short explain the rationale behind the order. (2 points ½ -1 page)

Question 4. (5 points 2+3)

a. Explain how tapping mode AFM works? How are height and phase images generated? (2 points 1-2 pages)
b. In detail explain how contrast is generated in SEM and TEM? (3 points 1 -2 pages)

Question 5. (5 points 3+2)

a. What is piezoelectric effect? Explain in detail how a quartz crystal microbalance senses mass. What kind of information one derives from frequency and dissipation changes during an adsorption study using QCM-D technique?(3 points 1-2 pages)
b. QCM-D technique gives wet mass while SPR gives dry mass. Explain this statement (1 point ½ page)
c. Three different surface treatments A, B and C result in Frequency changes 300Hz, -100Hz and 200Hz. What is the order of these surface treatments with respect to the extent of adsorption taking place on the surface? (1 point ½ page)

Question 6. (5 points 3+2)

a. Classify the following into destructive and non-destructive techniques. (1 point)
   Thermogravimetric analysis
   Raman Spectroscopy
   AFM
   CHNS analysis
b. Compare the elemental analysis techniques XPS and 'CHNS' analysis. What are advantages and dis-advantages of these techniques? (2 points 1-1½ pages)
c. What are the different types of chromatography? In short explain how each type of chromatography achieves solute separation. (2 points 1-2 pages)

Question 7. (5 points 1+1+3)

a. Explain in short the necessary condition for a nucleus to be NMR active. (1 point ½ page)
b. Explain the effect of strength of magnetic field on chemical shift. (1 point ½ page)
c. What are the components of a NMR spectrometer? Explain the terms Locking and shimming. What kind of solvent is used for NMR sample preparation and why? (3 points 1-2 pages)