Loughborough University

22MPP567

Advanced Materials Characterisation

Semester 1 2022/23 Online Short-window Exam paper

This is an online short-window examination, meaning you have a total of **2 hours plus an additional 30 minutes** to complete and submit this paper. The additional 30 minutes are for downloading the paper and uploading your answers when you have finished. If you have extra time or rest breaks as part of a Reasonable Adjustment, you will have further additional time as indicated on your exam timetable.

It is your responsibility to submit your work by the deadline for this examination. You must make sure you leave yourself enough time to do so.

It is also your responsibility to check that you have submitted the correct file.

Exam Help

If you are experiencing difficulties in accessing or uploading files during the exam period, you should contact the Exam Helpline. For urgent queries please call **01509 222900**.

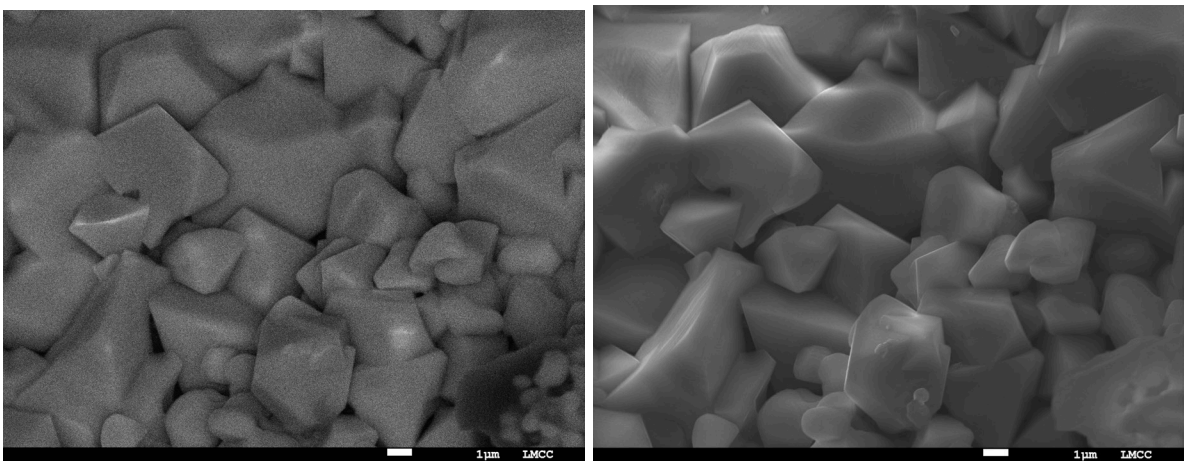
For other queries email examhelp@lboro.ac.uk

You may handwrite and/or word process your answers, as you see fit.

You may use a calculator for this exam.

Answer **THREE** questions.

1. A scanning electron microscope (SEM) is used to examine the surface morphology of an oxide formed on duplex stainless steels. Figure Q1 shows the SE images obtained by different SE detectors under same accelerating voltage (20KV) and working distance (10mm).



(i) (ii)

Figure Q1 (i) and (ii): Secondary electron (SE) images obtained using different SE detectors.

(a) Which SE detectors were used in obtaining Figure Q1 (i) and (ii) respectively? With an aid of a schematic diagram, justify your answer by comparing the detectors' positions, and types of electrons collected in each case. [10 marks]

(b) Explain the effect of following operation parameters within a scanning electron microscope (SEM) on the SE image obtained:

(i) Accelerating voltage; [3 marks]

(ii) Working Distance; [2 marks]

(iii) Aperture size. [2 marks]

(c) What ions are used in focussed ion beam (FIB) instruments? Give an application of FIB and justify why the ion species you have suggested are used for the suggested application.

[3 marks]

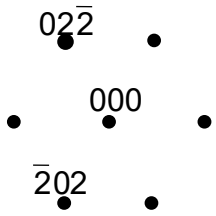
2. (a) With an aid of schematic ray diagram, describe how electron diffraction patterns are formed in transmission electron microscope (TEM). [4 marks]

(b) In an X-ray powder diffraction experiment the following reflections are observed (2θ):

29.99°
34.77°
49.99°
59.41°

The X-ray wavelength is 0.154 nm and the specimen has a fcc crystal structure. Calculate the lattice parameter? [8 marks]

(c) Sketch and index the following diffraction pattern



[2 marks]

(d) Calculate the Bragg angle (2θ / °) for the {111} planes of copper (a = 0.362 nm), using

(i) X-ray diffraction with Cu K_α radiation of λ = 0.15418 nm and

(ii) Electron diffraction with λ = 0.00251 nm. [2 marks]

(e) Compare the FOUR differences between X-ray and electron diffraction.

[4 marks]

Useful formula for Question 2

Bragg's law	$2d\sin\theta=n\lambda$
Interplanar spacing	$d = \lambda L/R$
Interplanar spacing	$d=\frac{a}{\sqrt{h^2+k^2+l^2}}$

3. You work within the QC department of a polymer manufacturer and your colleagues in the production department have asked you for some help.

(a) Your colleagues consistently use two polymers that are visually indistinguishable and need to quickly tell the difference to allow the waste to be directed back to correct stream for recycling. What technique would you suggest they use? Explain your reasoning.

[2 marks]

(b) They then want to determine whether their manufacturing process is consistent and reproducible. To do this they need to monitor molecular weight and dispersity, end group functionality and particle size. State three questions that you would ask to allow you to determine the correct methods and conditions to use, explaining what information would be obtained by asking these. [6 marks]

(c) The ^{1}H NMR spectrum of a monomer shown in Figure Q3.

(i) State how the integrals of the peaks are useful for molecular structure assignment. [1 mark]

(ii) Explain which peaks would be present in both monomers [2 mark]

(iii) Of the two chemical structures given (A and B) state which matches best to the features of the NMR spectrum observed.

[1 mark]

(iv) Explain your reasoning, considering the differences in chemical structure between the two monomers. [4 marks]

(v) Describe how the ^{1}H NMR spectra of the polymers formed from these monomers would differ, explaining why the changes occur. [4 marks]

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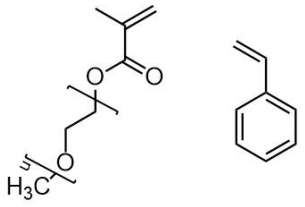
Q3 Continued/…



A B

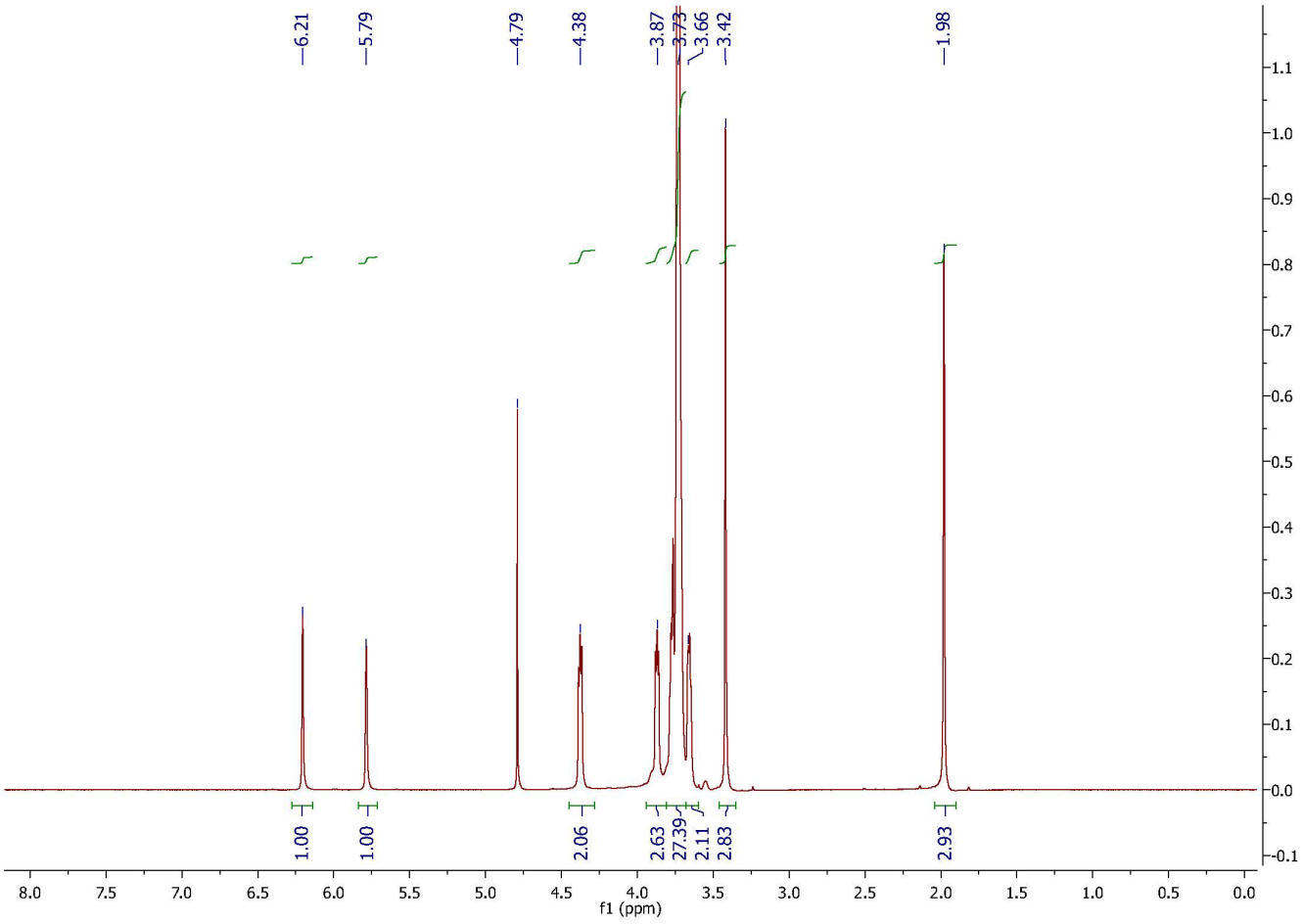


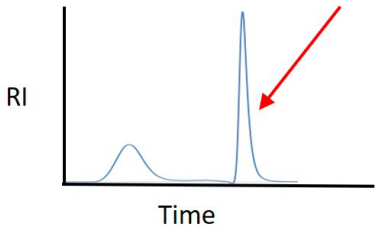
Figure Q3 – Chemical structures and ^{1}H NMR of monomer.

4. Size exclusion chromatography (SEC) is used to separate polymers based on their molecular weight.

(a) Explain how the interaction of functional groups in the polymers with the column materials is avoided. [2 marks]

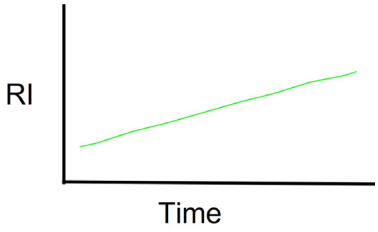
(b) Consider the size exclusion chromatography (SEC) traces below. State what may cause the issues highlighted by the arrows or described in (i) – (iv) and suggest how these could be rectified in practice.

(i)



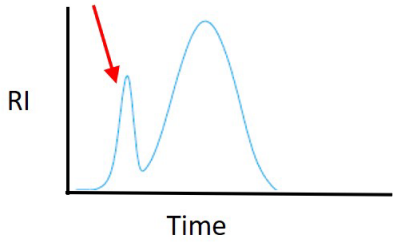
[2 marks]

(ii)



[2 marks]

(iii)



[2 marks]

Continued/…

(iv) No response in the differential refractive index (DRI) detector seen regardless of concentration of polymer. [2 marks]

(b) (i) In dynamic light scattering (DLS) analysis particle size is determined using the Stokes-Einstein equation. Use this to determine the hydrodynamic diameter of a polybutylmethacrylate particle in solvent X at 25°C given the following information:

$$Dh = kT/3\pi\eta_s D$$

$k = 1.38 \times 10^{-23}$ m^2 kg s^{-2} K^{-1}

η_s= solvent viscosity

$D = 8.1 \times 10^{-12}$ m^2/s

Dynamic Viscosity of solvent X at 25°C 3.6 mPA.s [3 marks]

(ii) Water is suggested as an alternative solvent as it is less viscous than Solvent X at the same temperature. Suggest what issues there may be when using different solvents for LS. [2 marks]

(iii) Light scattering can also be used to analyse the size of linear polymers in solution. Explain why the concentration of these polymers is important referring to the Stokes-Einstein equation above. [3 marks]

(c) Transmission electron microscopy (TEM) is required to help confirm the findings from the scattering techniques. Suggest what a suitable sample support and preparation method to analyse polymer nanoparticles, explaining your reasoning. [2 marks]

END OF PAPER

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