IMPERIAL COLLEGE LONDON

M.Eng EXAMINATION IN CHEMICAL ENGINEERING 2018

PART III and IV

and

M.Sc. in ADVANCED CHEMICAL ENGINEERING

For Internal Students of Imperial College London

This paper is also taken for the relevant examination
for the Associateship

PRODUCT CHARACTERISATION

Tuesday 8th May 2018: 10:00 - 11:30

Answer all four questions

**THIS EXAMINATION PAPER HAS SIX PAGES IN TOTAL
WHICH INCLUDES THIS COVER SHEET**

Before starting, please make sure that the paper is complete. Ask the invigilator for a replacement if your copy is faulty

Question 1 carries 30 marks
Question 2 carries 30 marks
Question 3 carries 40 marks

TURN OVER FOR QUESTIONS

Question 1 **[30 marks]**

(a) A start-up aiming to manufacture a new type of membrane has managed to synthesize a kilogram of the material (Material ICL) used to form the membrane. This material is micro/mesoporous and polymeric. Using the data presented in Table 1:

(i) Describe the main characteristics of Materials 1, 2 and 3.

(ii) Identify if Material ICL corresponds to Material 1, Material 2 or Material 3. Justify your answer.

[10 marks]

(b) (i) Sketch the thermogravimetric curve of Material ICL (atmosphere: N_2). Justify your answer.

(ii) Explain the origin of any weight drop or gain on the curve.

[6 marks]

(c) Material ICL could also be used as an adsorbent. To allow its use in adsorption applications, Material ICL would have to be pelletized. For this, it is mixed with an inorganic binder, *e.g.* alumina.

(i) Sketch the thermogravimetric curve of the pellet (atmosphere: N_2).

(ii) Explain any changes compared to the thermogravimetric curve of pure Material ICL. Justify your answer.

[6 marks]

(d) Formation of macropores can be expected during pelletization.

(i) What technique would you use to measure the volume of macropores in the pellets?

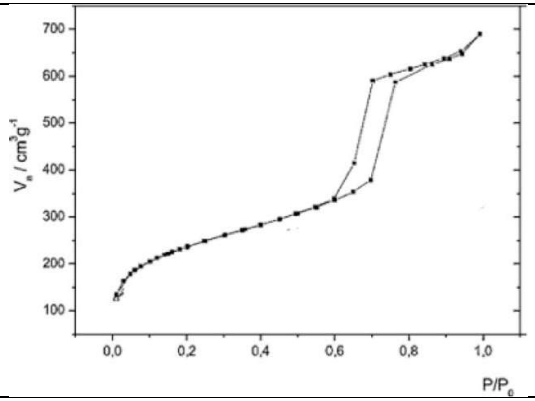
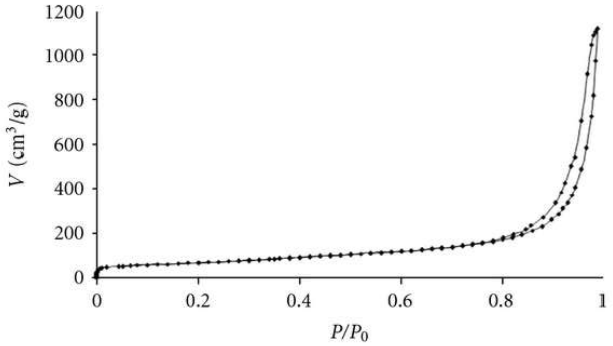
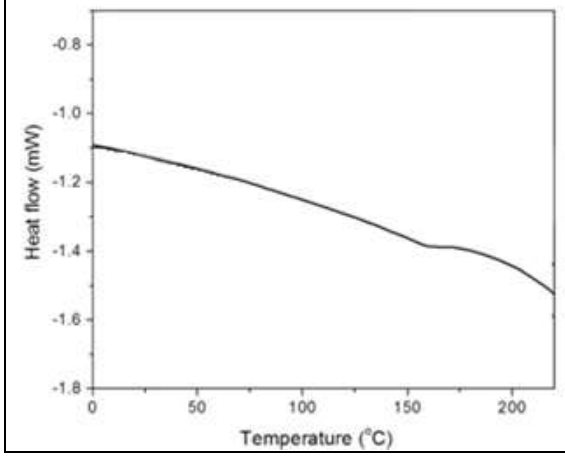
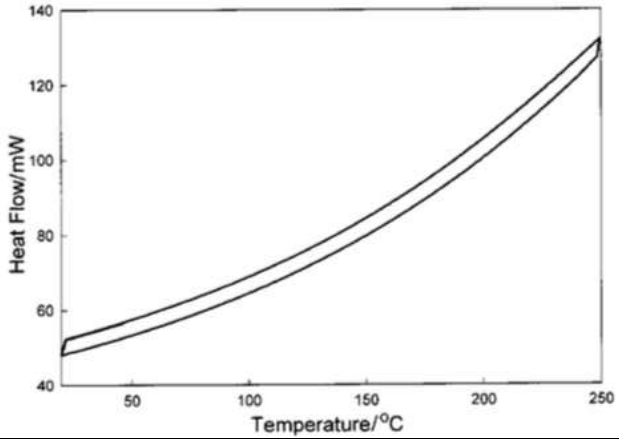
(ii) Summarise the principle of this technique and its limitations. [3 marks]

(iii) How would the pore diameter be calculated? [3 marks]

[8 marks]

Table for Question 1:

Table X. Summary of characterisation analyses and key features of three materials (Graphs adapted from: *Appl. Catal. B* 146 (2014) 112-122; *J. Mater. Chem.*, 8, (1998) 1205-1211; *Nature Comm.* 4, Article number 1357 (2013)).

Material 1	**Material 2**	**Material 3**
Pore size range goes from 5 Å to 30 Å.		
	No glass transition observed.	

TURN OVER

Question 2 **[30 marks]**

You are designing a new product for the food industry, crunchy mayonnaise, consisting of bulgur wheat and mayonnaise. The bulgur wheat consists of small particles of the wheat which are 2 mm in diameter and have a density of 1200 $kg.m^{-3}$. The mayonnaise consists of 70% by mass olive oil (density 915 $kg.m^{-3}$ viscosity 5.6 mPas) and 30% by mass of an aqueous phase (containing eggs and vinegar; density 1050 $kg.m^{-3}$; viscosity of 3.5 mPas). The mayonnaise is in the form of an oil in water (aqueous phase) emulsion.

(a) Outline how you would measure the droplet size of the oil in this emulsion

[4 marks]

(b) Calculate the volume fraction, ϕ, of oil in the emulsion

[2 marks]

(c)

(i) Assuming the Einstein relation, estimate the viscosity of the emulsion

(ii) Now use the Kreiger - Dougherty relationship (below) to estimate the viscosity, taking the maximum particle packing volume fraction, ϕ_{max} to be 0.75 and the intrinsic viscosity $[\eta]$ to be 2.5.

(iii) Why are the values calculated in (i) and (ii) different; which value for the viscosity is likely to be closer to the experimental value?

(iv) Estimate the sedimentation rate of the bulgur wheat particles in this mayonnaise.

[16 marks]

(d) To be a successful product sedimentation needs to be eliminated completely.

(i) How may this be achieved?

(ii) Quantify the minimum rheological property(ies) the mayonnaise must have in order to prevent sedimentation

[8 marks]

Formulas for Question 2:

$$\eta_{rel} = \frac{\eta}{\eta_0} = \left(1 - \frac{\phi}{\phi_{max}}\right)^{-\phi_{max}[\eta]}$$

where η is the viscosity of the mayonnaise and η_0 is the viscosity of the continuous phase of the emulsion

Question 3 **[40 marks]**

(a) Which of the following four molecules may show (i) Infrared absorption spectra? (ii) Raman spectra? $C_6H_5CH_3$, $^{79}Br^{81}Br$, NH_3 and H_2. Briefly explain why.

[10 marks]

(b) Explain how would you spectroscopically measure polymer laminates and semi-quantitatively assess spatial resolution in z-direction to the plane of the laminates using micro ATR-FTIR spectroscopic imaging. Support explanations with corresponding equations and/or schematics as appropriate.

[10 marks]

(c) Describe and explain the principles of tip-enhanced Raman spectroscopy (TERS) and microscopy. Explain what spatial resolution can be achieved with this technique in analysis of nanomaterials and provide some examples of applications. Support explanation with corresponding schematics as appropriate.

[10 marks]

(d) How would you measure distribution of substances in microfluidics flows using FTIR spectroscopic imaging? Provide examples of relevant applications. Describe how the fields of view (measured areas) and depths are analysed in this approach. Support your explanation with corresponding equations and schematics as appropriate.

[10 marks]

END OF PAPER