UNIVERSITI SAINS MALAYSIA

First Semester Examination
Academic Year 2005/2006

November 2005

KAA 507 – Surface and Thermal Analysis

Time : 3 hours

Please make sure this paper consists of FOURTEEN printed pages before answering the questions.

Answer any FIVE questions.

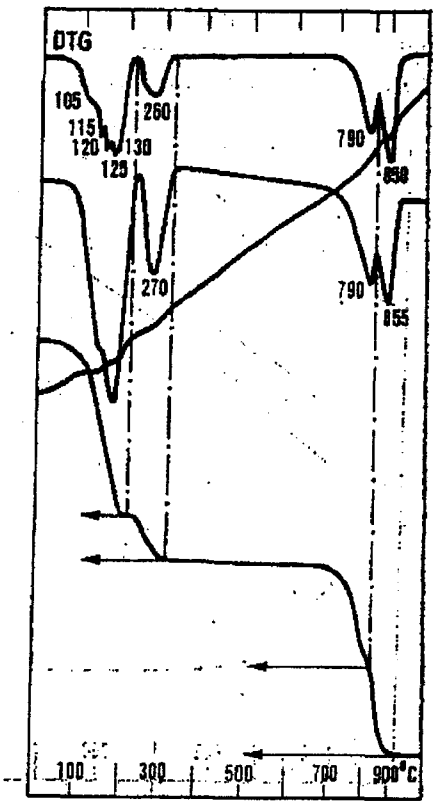
Only the first five questions answered in the answer book will be marked.

1. (a) (i) What is thermal analysis and explain how could this technique can be regarded as an analytical method?

(ii) What are the basic components for thermogravimetric analysis instrument (TGA) and type of information can you get from a TG and its derivatives curves (DTG)?

(5 marks)

(b) In the figure given below are three thermogram corresponding to TG, DTGA and differential thermal analysis (DTA) for hydrate copper sulphate ($CuSO_4.5H_2O$). Label and explain each of the thermogram and also explain why DTGA more sensitive as compared to DTA?



(5 marks)

(c) X-ray photoelectron spectroscopy (XPS) is a surface analytical technique with the capability to measure the binding energy variations of an atom in its chemical environment.

(i) Explain briefly the basic principles of XPS with emphasis on the source of irradiation, the nature of sample, detection of the generated signal and the pattern of the XPS spectrum.

(ii) What is meant by the chemical shift in XPS? Describe briefly, with an example, how chemical shift can be applied in determination of molecular structure?

(iii) Describe the advantages of XPS compared to Auger electron spectroscopy (AES).

(10 marks)

2. (a) Describe briefly the principle differences and similarity between differential thermal analysis (DTA) and differential scanning calorimetry (DSC) in term of instrument and interpretation of the results? Explain the factors that you think may contribute to the reproducible results.

(5 marks)

(b) Explain briefly the difference between heat-flux and power- compensated DSC? State all information you would expect to obtain from the DSC thermogram for an organic polymer.

(5 marks)

(c) Compositional analysis by means of scanning electron microscopy with X- ray energy dispersive mode (SEM-EDX) is considered as a qualitative analysis.

(i) Describe briefly on how the X- ray is generated, detected and analysed in SEM-EDX.

(ii) Explain, with diagram, THREE type of artefacts or false X- ray signal that commonly occurred in SEM-EDX analysis.

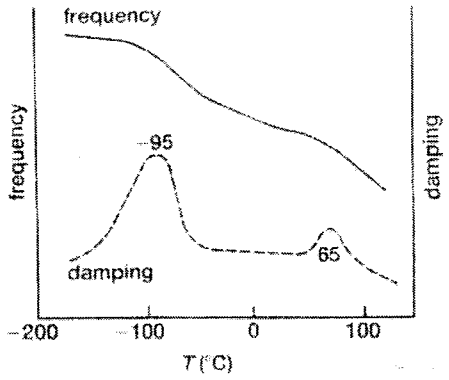
(iii) Describe clearly why electron probe microanalysis (EPMA) is considered a more quantitative analysis technique compared to SEM-EDX

(10 marks)

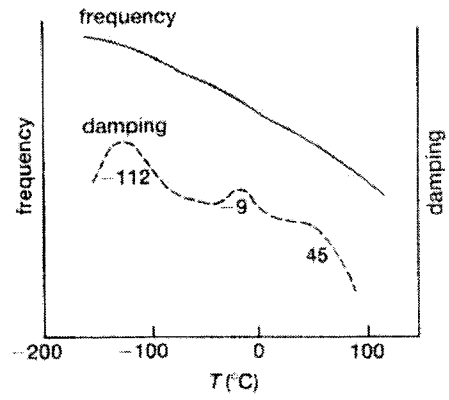
3. (a) (i) Explain the difference between absolute and difference measurement for a thermodilatometry (TD). Describe the principle difference and advantages between of laser dilatometer and optoelectronic transducer when is used as transducer in TD.

(ii) Describe briefly the applications of thermodilatometry (TD) which are widely used in the industry and give two examples.

(iii) Given a dyamic mechanical analysis(DMA) results of two different samples of polyethylene in the chart below. Explain the properties of the two polyethylene.



(a) Linear polyethylene



(b) Branched polyethylene

(10 marks)

(b) A crystalline solid sample consists of a mixture of iron oxides (Fe_3O_4, Fe_2O_3 and FeOOH).

(i) Explain briefly the surface analytical technique that is required to determine the crystal structure of each of the iron oxides.

(ii) Describe the analytical techniques to determine quantitatively the surface chemical composition of the iron oxides.

(10 marks)

4. (a) The following data list volumes of ammonia gas adsorbed (reduced to S.T.P) per gram activated charcoal at 0 °C.

Pressure/mm Hg	50	100	200	400	600
Volume/ $cm^3 g^{-1}$	74	110	150	177	189

Show that the data fit a Langmuir adsorption isotherm expression and evaluate the constants. Calculate the specific surface area of the activated charcoal, if the cross section molecular area of nitrogen is 16.2 $Å^2$.

(6 marks)

(b) State four criteria that are used in distinguishing physical or chemical adsorption. Draw in the same diagram the schematic potential energy curve for (i) physical adsorption and (ii) chemical adsorption of a diatomic gas X_2 onto a surface of a metal M. Indicate the activation energy of chemisorption. Justify the shapes and relative position of the curves.

(7 marks)

(c) Secondary Ion Mass Spectroscopy (SIMS) is one of the most sensitive and specific surface analytical technique.

(i) Describe the basic principles of SIMS with emphasis on the production and detection of the secondary ion and the shape of SIMS spectrum.

(ii) Discuss why quantification of SIMS spectrum difficult to measure.

(iii) How is the depth profiling analysis carried out in SIMS? What are the advantages of depth profiling analysis in SIMS as compared to XPS?

(10 marks)

5. (a) Based on the BET (Brunauer, Emmett and Teller) model of adsorption when limiting number of adsorbed molecular layers is limited to the number n, at saturation , the BET treatment leads to the modified equation,

$$\frac{X}{X_m} = \frac{c(p/po)}{1- p/po} \cdot \frac{1-(n+1)(p/po)^n + n(p/po)^{n+1}}{1+(c-1)(p/po) - c(p/po)^{n+1}}$$

Where X is the amount adsorbed at relative pressure p/po , X_m is a monolayer capacity, n is the of adsorbed molecular layers and c is a constant. Show that this equation can account for the standard BET equation and Langmuir adsorption isotherm.

(7 marks)

(b) The following data refer to the adsorption of nitrogen on activated carbon from rice husk at -196°C:

Relative Pressure	Vol. adsorbed ($cm^3 g^{-1}$)
0.001	61.869
0.005	66.336
0.015	70.235
0.041	73. 371
0.102	76.072
0.200	77.829
0.311	79.120
0.430	80.103
0.700	81.714
0.900	84.101
0.967	86.623
0.994	88.928
0.944	87.352
0.735	84.668
0.491	83.024
0.411	81.904
0.300	81.151
0.241	80.723
0.109	79.054

(i) Calculate the monolayer capacity (X_m) by point B, BET and single point methods.

(ii) Use the BET value to calculate a specific surface area for above activated carbon. Compare this value with the other two methods, taking the molecular cross section area of nitrogen as 16.2 $Å^2$.

(iii) Give the type of the isotherm obtained and explain briefly about the nature and pore shape of this activated carbon.

(13 marks)

6. (a) What are the principle differences between surface area measurement by gas adsorption and mercury penetration? Explain the accuracy limits in both methods. State two other methods that are used in surface area determination.

(7 marks)

(b) Explain briefly, by providing two examples, the importance of evolved gas detection (EGD) and evolved gas analysis (EGA) which couple with basic thermal analysis. Describe briefly advantages and disadvantages of both the methods.

(6 marks)

(c) (i) Explain briefly , what is the viscoelastic behaviour?

(ii) What is the effect of temperature on stress-strain curve for a viscoelastic sample? Give an example .

(7 marks)

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