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APPENDICES

APPENDIX A

Instrumentations

A1 **UV-Vis Spectrophotometer** (Harvey, 2000)

The instrument used in ultraviolet-visible spectroscopy is called an UV/vis spectrophotometer. It measures the intensity of light passing through a sample (I), and compares it to the intensity of light before it passes through the sample (I_o). The ratio I / Io is called the transmittance, and is usually expressed as a percentage (%T). The absorbance, A, is based on the transmittance:

$$A = -\log (\%T)$$

The basic parts of a spectrophotometer are a light source (often an incandescent bulb for the visible wavelengths, or a deuterium arc lamp in the ultraviolet), a holder for the sample, a diffraction grating or monochromator to separate the different wavelengths of light, and a detector. The detector is typically a photodiode or a CCD. Photodiodes are used with a monochromator, which filter the light so that only light of a single wavelength reaches the detector. Diffraction gratings are used with CCDs, which collects light of different wavelengths on different pixels.

Figure A1 Agilent Model 8453 spectrophotometer; an UV-visible spectrophotometer

A spectrophotometer can be either single beam or double beam. In a single beam instrument (such as the Spectronic 20), all of the light passes through the sample cell. Io must be measured by removing the sample. This was the earliest design, but is still in common use in both teaching and industrial labs.

In a double-beam instrument, the light is split into two beams before it reaches the sample. One beam is used as the reference; the other beam passes through the sample. Some double-beam instruments have two detectors (photodiodes), and the sample and reference beam are measured at the same time. In other instruments, the two beams pass through a beam chopper, which blocks one beam at a time. The detector alternates between measuring the sample beam and the reference beam.

Samples for UV/Vis spectrophotometry are most often liquids, although the absorbance of gases and even of solids can also be measured. Samples are typically placed in a transparent cell, known as a cuvette. Cuvettes are typically rectangular in shape, commonly with an internal width of 1 cm. (This width becomes the path length, L, in the Beer-Lambert law.) Test tubes can also be used as cuvettes in some instruments. The best cuvettes are made of high quality quartz, although glass or plastic cuvettes are common. (Glass and most plastics absorb in the UV, which limits their usefulness to visible wavelengths.).

A2 **Gas chromatography/Mass spectrometry (GC/MS)** (Robinson, 2005)

The GC-MS is composed of two major building blocks: the gas chromatograph and the mass spectrometer. The gas chromatograph utilizes a capillary column which depends on the column's dimensions (length, diameter, film thickness) as well as the phase properties (e.g. (5% (phenyl) polysiloxane)). The difference in the chemical properties between different molecules in a mixture will separate the molecules as the sample travels the length of the column. The molecules take different amounts of time (called the retention time) to come out of (elute from) the gas chromatograph, and this allows the mass spectrometer downstream to capture, ionize, accelerate, deflect, and detect the ionized molecules separately. The mass spectrometer does this by breaking each molecule into ionized fragments and detecting these fragments using their mass to charge ratio. These two components, used together, allow a much finer degree of substance identification than either unit used separately. It is not possible to make an accurate identification of a particular molecule by gas chromatography or mass spectrometry alone. The mass spectrometry process normally requires a very pure sample while gas chromatography using a traditional detector (e.g. Flame Ionization Detector) detect multiple molculears that

happen to take the same amount of time to travel through the column (*i.e.* have the same retention time) which results in two or more molecules to co-elute. Sometimes two different molecules can also have a similar pattern of ionized fragments in a mass spectrometer (mass spectrum). Combining the two processes makes it extremely unlikely that two different molecules will behave in the same way in both a gas chromatograph and a mass spectrometer. Therefore when an identifying mass spectrum appears at a characteristic retention time in a GC-MS analysis, it typically lends to increased certainty that the analyte of interest is in the sample.

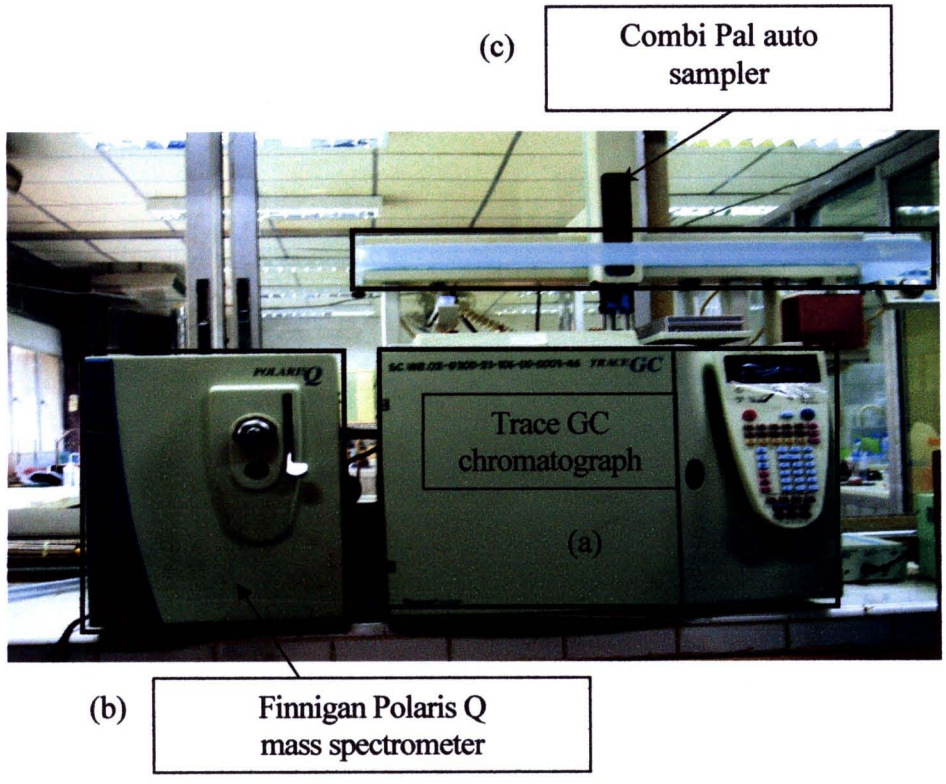

Figure A2 The photograph of GC-MS instrument used in this study (a) Trace GC chromatograph (b) Finnigan Polaris Q mass spectrometer (c) Combi Pal autosampler

APPENDIX B

Research publications

RESEARCH PUBLICATIONS

1. Supattra Supakarn, Wimonrat Tongpoothron, Saksit Chanthai, Chalerm Ruangviriyachai. **Purification of glycerine residue from transesterification of *Jatropha curcas* oil.** Poster presentation for The 6th International Congress on Chemistry for Innovation (PERCH-CIC Congress VI) May 3-6, 2009; Jomtien Palm Beach Hotel & Resort, Pattaya, Chonburi, Thailand, p. 162.
2. Supattra Supakarn, Wimonrat Tongpoothron, Saksit Chanthai, Chalerm Ruangviriyachai. **Improvement of a process for the purification of glycerine residue from transesterification of *Jatropha curcas* oil.** Poster presentation for The Pure and Applied Chemistry International Conference 2010 (PACCON 2010); January 21-23, 2010; Sunee Grand Hotel and Convention Center, Ubon Ratchathani University, Ubon Ratchathani, Thailand, p. 52-55.

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