CHEMICAL DOSIMETRY EXPERIMENT USING UV-VIS SPECTROPHOTOMETER

CENTER FOR MEDICAL AND RADIATION PHYSICS

Aim:

- To prepare Ferrous ammonium sulfate, Benzoic acid, and Xylenol orange (FBX) Chemical dosimeter.
- To study the absorbance spectrum, i.e. wavelength vs absorbance of FBX dosimeter solution with the help of a UV/Vis spectrophotometer
- To calibrate the UV/Vis spectrophotometer in terms of absorbed dose using FBX dosimeter solution and find out the unknown dose to a sample.

Equipments:

- Linear Accelerator
- UV-VIS Double Beam Spectrophotometer (IG-28DS)
- Single Distilled water
- 250mL Standard Volumetric Flask
- Pipette
- Precision Balance
- Polypropylene irradiation tubes
- Lint-free tissue papers
- Optical quartz cuvettes with 10 mm pathlength
- Reagents (Analytical grade):

(i) Ferrous ammonium sulfate [$Fe\ (NH_4)_2\ (SO_4)_2.\ 6H_2O$]
(ii) Benzoic Acid (C_6H_5COOH)
(iii) Xylenol Orange ($C_{31}H_{32}N_2O_{13}S$)
(iv) Sulphuric Acid (H_2SO_4)

Theory:

A dosimeter that determines dose by measuring chemical changes induced in materials by ionizing radiations is known as a chemical dosimeter. One such chemical dosimeter is a dilute solution of ferrous ammonium sulfate in dilute sulphuric acid containing benzoic acid and a dye xylenol orange. This dosimeter, also known as FBX (Ferrous ammonium sulfate-benzoic acid-xylenol orange), can measure doses in the range of 0.2 to 30Gy, with an uncertainty less than 0.9%.

The principle behind the FBX dosimeter is the measurement of absorbed dose by measuring the change in absorbance of the solution. When this solution is exposed to ionizing radiation, Ferrous ions (Fe^{2+}) get oxidized to Ferric (Fe^{3+}) ions. This Ferric ion forms a 1:1 complex with xylenol orange (Fe^{3+}- XO complex), and xylenol orange has an absorption peak in the visible region of the electromagnetic spectrum (500 - 550 nm). The concentration of Ferric ions increases with the amount of radiation dose, thereby increasing the absorbance of the solution. This change in absorbance can be used to estimate the Absorbed Dose. The presence of Benzoic acid increases the sensitivity of the solution by increasing the radiation chemical yield of the Ferric ions.

The Calibration of the UV/Vis spectrophotometer in terms of absorbed dose can be interpreted as establishing a relationship between absorbance and radiation absorbed dose. This relationship can be established by exposing the solution to known radiation doses and measuring the absorbance at the peak for each dose value. When we plot this absorbance versus absorbed dose, we will get a calibration curve. This calibration curve can be used to estimate the unknown doses.

To ensure the accuracy and reliability of this curve, it is essential to validate it. Validation can be done by measuring a known dose of an irradiated sample. The measured dose can be compared with the delivered dose, and an uncertainty of less than 0.9% shows a reliable and accurate calibration curve. Further unknown doses can be calculated using the equation derived from this calibration curve,

Components of UV-Vis Spectrophotometer (IG28-DS):

- **Light Source:** A hydrogen discharge lamp or deuterium lamp (D2 Lamp) is used as a UV source and a tungsten filament lamp (T Lamp) is used for visible light source.
- **Monochromator:** The light source emits polychromatic light so to make it monochromatic and to have a desired range of wavelengths, monochromators are used. Diffraction gratings are used to separate light of different wavelengths with high resolution.
- **Beam splitter:** It splits the beam into two parts perpendicular to each other. The beam splitter reflects half of the light and transmits the other half, providing us with two sources (One for the reference sample and the other for the exposed samples).
- **Sample Holder (Cells or Cuvettes):** The liquid samples must be kept in a container that should be transparent to UV and visible radiation. These specially designed containers or holders are called Cuvettes. Cuvettes made of quartz are used for both UV and Visible regions, whereas glass cuvettes are more suitable for visible light. The standard optical path length of these cuvettes is usually 1 cm.
- **Detectors:** High-sensitive photodiodes convert the incident photons into an electric current.
- **Display/Recorder:** The attached display/recorder records the transmittance or absorbance as a function of the wavelength of the incident radiation beam.

Figure 1: Microprocessor UV-VIS Double Beam Spectrophotometer (IG-28DS)

Procedure:

Preparation of FBX dosimeter:

500mL of FBX solution is composed of the following compounds:

Ferrous ammonium sulfate	0.2mol/m^3
Benzoic Acid	5.0mol/m^3
Xylenol orange	0.2mol/m^3
Sulphuric Acid	40mol/m^3

In singly distilled water.

1. **Preparation of 0.05N H_2SO_4 solution (250mL):**
   Transfer 340 µL of Analytical Reagent grade sulfuric acid (sp. Gravity 1840 kg m^{-3}) in a clean, 250mL standard volumetric flask containing distilled water of around 100mL. Stopper the flask and mix well.

2. **Preparation of dosimetric solution:**
   Weight 39.214 mg (0.1mM) of ferrous ammonium sulfate [Fe $(NH_4)_2$ $(SO_4)_2$ $.6H_2O$], on a precision balance and transfer it carefully to a clean flask containing about 100 mL of 0.05N H_2SO_4 stock solution. Weight 305.3mg (2.5mM) of Benzoic acid and transfer it carefully to the flask. Then add 67.3mg (0.1mM) of xylenol orange to it. Make up the volume with the H_2SO_4 stock solution and stir it well so that all the solutes mix well in the flask. Add distilled water to it so that the total volume of the solution will become 250mL. Stopper the flask and shake it well to dissolve the contents completely and carry out aeration of the solution. The benzoic acid will take time to dissolve, so it is recommended that the solution be heated after adding it for quick mixing.

3. **Preparation of Dosimeters**
   Take 7 pre-cleaned polypropylene tubes. Rinse each tube with Distilled water and then with FBX solution at least three times before filling it. Care should be taken not to touch the inside of the tube or stopper while filling it. It should also be ensured that no air bubble is trapped inside while stoppering the tubes. Wipe out the outsides of the tubes with clean tissue paper and number them.

Irradiation Setup:

- Place the plastic tube containing the FBX dosimeter in between two 1cm Gel boluses (normally used in radiotherapy treatment), which act as a build-up and backscatter for the dosimeter.
- Irradiate the samples to a known amount of radiation doses (E.g. 2Gy, 4Gy, 6Gy, etc.).
- To ensure uniform irradiation, the samples should be exposed equally from the Anteroposterior (AP) and Posteroanterior (PA) directions.
- Similar procedures should be followed for all the samples.

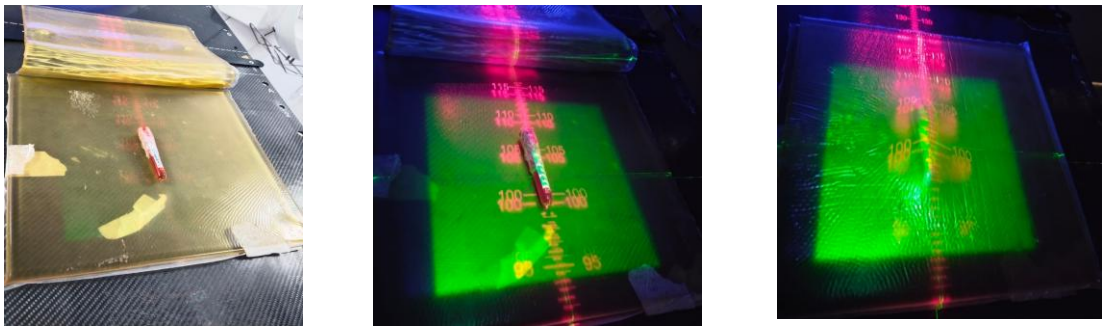

Figure 2: irradiation setup- FBX dosimeter placed in between 1cm Gel Bolus

Measurement Setup:

- Power On the UV/VIS spectrophotometer and allow it to stabilize for 15 to 20 minutes.
- Before inserting the quartz cuvette into the spectrophotometer, perform an air scan (no cuvettes inside, i.e. both the sample and the reference compartment are empty) to check the machine's performance.
- Take two cuvettes filled with unirradiated FBX solution and perform the blanking process. This blanking ensures that, the baseline is set to Zero for each wavelength value.

- Remove the cuvette from the sample compartment, wash it properly, and fill it with the irradiated FBX solution. Place it back in the sample compartment after cleaning the outside part properly with the help of lint-free tissue paper.
- Perform a complete range of wavelength scan and save the resulting absorbance-wavelength spectrum.
- Repeat the above procedure for different irradiated solutions and save them with suitable nomenclature.
- From these spectrums, note the wavelength at which the maximum absorption peak occurs.
- Tabulate the dose and absorbance value at the peak, and plot these values in a graph. A reference graph is shown in Figure 2. Derive the equation from the graph and use it to determine the unknown dose values.
- To check the repeatability, repeat the measurement for a known dose sample. Calculate the dose value from the absorbance by using the derived equation. Compare this calculated value with the delivered dose.
- For more detailed operation of the UV/VIS spectrophotometer software, refer to the software manual.

Tabulation:

Sl.No	Wavelength	Dose (Gy)	Absorbance
1	545 nm	2	0.1873
2		2	0.1970
3		4	0.3611
4		6	0.52
5		8	0.675
6		10	0.8246

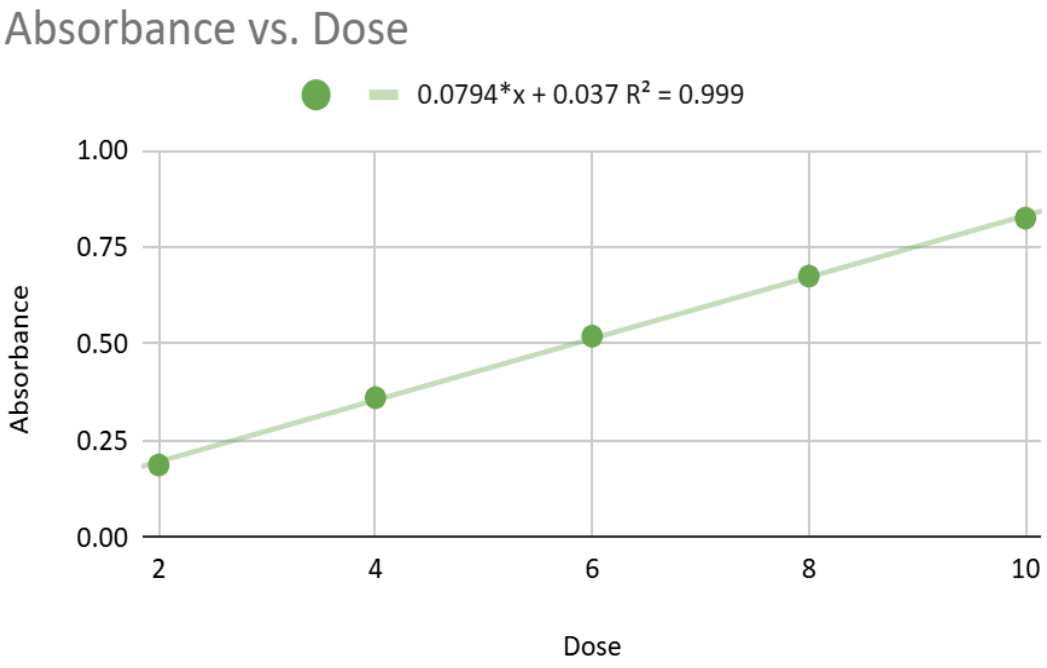

Figure 3: Plot of Dose Vs absorbance data for FBX solution

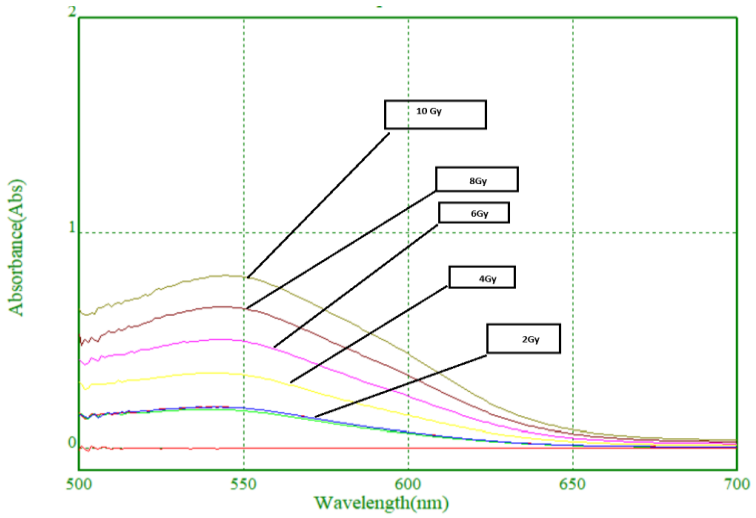

Figure 4: Absorbance spectrum for Fe^{3+} ion in standard FBX solution

The plot of Dose vs. absorbance was found to be a straight line given by the equation

$$Y = 0.0794X + 0.037$$

Validation of Unknown Dose:

Absorbance From unknown sample= 0.1970

Dose of Unknown Sample:

$$Dose = \frac{Absorbance\ -\ 0.037}{0.079} = \frac{0.1970 - 0.037}{0.079} = 2.02Gy$$

Actual Dose given (True Dose) = 2Gy

$$\%\ \text{Error} = \frac{Measured\ Dose - True\ Dose}{True\ Dose} X\ 100 = \frac{2.02-2}{2} X\ 100 = 1\%$$

Determination of Unknown Dose:

Result:

Conclusion:

Precaution:

- Always wear gloves while preparing and handling the chemical dosimeter.
- Add sulphuric acid into water because it will dissipate the heat due to the exothermic reaction of sulphuric acid with water.
- Always be careful while mixing the solute into the solvents as it may get spread out to your cloth or ground due to mishaps.
- Always use lint-free tissue paper to clean the cuvettes.
- Be careful while placing the cuvette into the compartments.
- While pouring irradiated solution into the cuvette, use a beaker below to avoid the chemical solution falling onto the table or floor.
- Always turn off the spectrophotometer lamps before switching them off.

References:

- FBX aqueous chemical dosimeter for measurement of dosimetric parameters by Manoj Semwal et.all - (41) FBX aqueous chemical dosimeter for measurement of dosimetric parameters | manoj semwal - Academia.edu
- Gupta, B.L., Kini, U.R., Bhat, R.M., Madhvanath, U., 1982. Use of the FBX dosimeter for the calibration of cobalt-60 and high-energy teletherapy machines. - pdf
- UV/VIS Analyst Software Manual