Chapter - 5

EXPERIMENTAL
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A. THERMOLUMINESCENCE GLOW CURVE READER

TL glow curve reader system used in the present investigation consisted of a photomultiplier (PM) tube Thorn EMI 9804 B, a high voltage DC supply unit, a DC amplifier, temperature programmer, X-Y and strip chart recorders. Block diagram of the unit is shown in fig 5.1. Weighed quantity of the sample was spread uniformly on a kanthal strip (an alloy of Fe-72%, Cr-23%, Al-3% and Co-2%) which acted as the heater. Size of the kanthal strip used in the present investigation was 30x15x.25mm with a rectangular recess of 2mm depth, 18x12mm length and breadth and a Chromel-Alumellel thermocouple spot-welded at the centre on the lower side of the kanthal strip. Because of the small size of the heater, high temperature can be attained with low power input and after switching off the input power supply the cooling is faster. Kanthal strip is secured tightly between two brass electrodes. The electrodes are fitted in 233x110x24mm backlite plates which can slide in and out of an aluminium guide box, at the top of which the EMI 9804 B photomultiplier tube is housed in a light tight brass cylinder. When the bakelite plate is fully inside the guide box, the sample position (heater) comes vertically under the photomultiplier and has nearly 100% efficiency for the collection of TL-emission. To reduce the thermal background of the heater, an aluminium cover, with a central hole of 20mm diameter was used.
FIG 5.1 BLOCK DIAGRAM OF TL SET-UP
TL Detection Unit
D.C.Supply Unit.
U.C. Amplifier.
X - Y Recorder

Temperature Programmer
D.C.Supply Unit.
D.C.Amplifier.

X - Y Recorder
over the sample so that the PM can see only the sample and not the heater strip. Further reduction of thermal background was achieved by introducing a quartz window between the heater and the photomultiplier. Sample could be changed by sliding the drawer half way out.

A negative voltage of 900 V was applied to the photomultiplier tube which detected the light output from the sample. Power to the heater plate was fed from the Indotherm-487 temperature programmer designed to heat the sample at any required rate ranging from 5°C/min to 1000°C/min. The light emitted by the sample on heating was detected by the photomultiplier tube and subsequently amplified and recorded using X-Y recorder. The heating rate was kept constant namely, 400°C per minute. The linearity and reproducibility were found correct within 2 to 4% for all TL measurements.

B. FURNACE FOR ANNEALING: Thermal annealing of the sample was carried out in a muffle furnace with sample holding space 20x8x8cm. The temperature was maintained ±1% using a temperature controller which supplied the required current to the furnace. This furnace was used to study the thermal effects on the zeolite samples.

C. BETA - SOURCE: A beta source Sr - 90 with a strength of 50 mCi was used during the experiments. Its irradiation rate was 200 and 2400 rads per minute at an irradiation distance of 14mm and 3mm respectively.
D. X-RAY DIFFRACTOMETRY:

The sample studied with the diffractometer was ground in an agate mortar. The grounded mineral was sieved through a sieve of size 80 microns. The sample holder was a rectangular sheet of aluminium 4x4 cm square and 1.5mm thick. It had a rectangular hole of 1.7x2 cm. Over the hole on one side of the plate a clean glass slide was placed and bound firmly over the hole with cellotape. The recess thus formed on the other side of the plate was tightly filled with powder specimen. The exposed surface of the specimen was levelled and the aluminium plate was then placed in its position in the machine. An excess of the sample powder is placed. The powder is tamped and compressed with another glass plate and levelled off. The surface of the powder is absolutely plain and the holder is then attached to the machine.

The diffractometer used was Philips model 1700. The alignment of the instrument was checked by a silicon standard. The instrumental conditions were: Cu K radiation; Ni filter. Operating voltage 40 KV, 32 mA. The goniometer was run at 5° per minute (20 drive).

E. The present experiments also involved thermogrameric analysis and differential thermal analysis measurements

i) Thermogravimetry (TG)

Several methods of analytical chemistry are based on measuring chemical or physical changes in a sample, when its
temperature is raised in a controlled manner. The result of such a measurement can often be expressed as graph of one specific physical property versus the temperature of the sample. In many cases the analysis of this curve can be done by the methods developed for other Thermally Stimulated Process such as TL or ITC. In thermogravimetry analysis (TGA) the mass (or rather the weight) of the sample, heated in an environment in a controlled manner, is recorded as a function of temperature or time. The TG curve is a stepwise curve in which each step indicates a process of either an increase or a decrease of the sample mass. Processes which causes increase of mass are adsorption and absorption of gases and vapours, and chemical reactions between the sample and gaseous reactants. Decrease of mass may be the result of sublimation of the sample, its dehydration, and chemical decompositions which releases gaseous products.

ii) Differential Thermal Analysis (DTA)

In the differential thermal analysis (DTA) technique, the difference between the temperature of the sample \( T_s \) and that of \( T_r \) as both the sample and reference are maintained in a furnace and heated up or cooled down. The reference sample is a material whose thermal properties (heat conduction and heat capacity) do not undergo abrupt changes in the temperature range of the measurement. Thus, the plot of \( \Delta T = T_s - T_r \) versus time or temperature is a nearly horizontal or a slowly changing line, unless abrupt enthalpic changes occur in the sample. Such changes can be
the result of endothermic or exothermic chemical reactions, phase transition etc. Each such enthalpic change usually appears as an upward or downward peak in the DTA curve. The analysis of these peaks, can sometimes be done using methods developed for TL and TG peaks. However, this must be done with care since the equations describing a TL peak are slightly different from the equations describing other TSP’s. Careless adoption of analysis methods which are useful in other areas of DTA curves may lead to erroneous results. For instance, the method of various heating rates which was proposed by Byissinger (1956,57) for evaluating activation energies of DTA peaks was later criticized by Reed et. al.(1965) who showed that, as it is, this method is not appropriate for DTA results.

In the present investigation Shimadzu DTA abd TGA experimental set-up were used in which the heating rate was maintained at 15 °C/min. the sample was heated in nitrogen atmosphere.