CENTRALIZED RESOURCE LABORATORY, UNIVERSITY OF PESHAWAR, PAKISTAN: FACILITATING MULTIDISCIPLINARY RESEARCH

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INTRODUCTION

Well-equipped research laboratories are the fundamental requirements of good educational institutions, where researchers can synthesize and analyze the beads of science. Research laboratories are so important today that we cannot separate teaching from research in any institution of higher learning. The status of a higher education institution is determined by the quality of research it conducts as well as the quality of the graduates it produces. To facilitate this to happen, the idea of establishing a modern well-equipped research laboratory pinched the academic and research community in the region and thus the idea was finally materialized with the establishment of centralized resource laboratory (CRL) at the premises of the Department of Physics, University of Peshawar (UOP). State of the art and latest equipments were procured and clustered at one location with the vision for their diverse use in basic and applied research in multiple areas of science. This clustering specifically aimed at facilitating the availability of the many highly costly testing and analytical instruments to many departments and research centres which normally were beyond their purchasing power on their own. This also in turn not only avoided the problem of duplication of such expensive equipments but it maximized their use and thus ensured quality research more cost-effective and economical.

Facilities

The equipments installed in the CRL that facilitate research, testing include the following:

- 200KV Transmission Electron Microscope (JM 2100, JEOL, Japan) with EDX (INCA100/ Oxford Instruments, UK)
- 30KV Scanning Electron Microscope (JSM5910, JOEL, Japan) with SEI and EDX detectors (INCA200/ Oxford Instruments, UK)
- Inverted Metallurgical Microscope (PMG-3, Olympus, Japan)
- X-ray Diffractometer (JDX 3532, JOEL, Japan) with CuKα source
- Universal Testing Machine (Extension, Compression, Bending) up to 100KN (Testometric UK. Model 500-100KN) with temperature range of -70 to 250°C
- Atomic Absorption Spectrometer (Analyst 700, Perkin Elmer USA) Mercury hydride system (MHS), Flame and graphite furnace techniques and 26 lamps available
- High Performance Liquid Chromatograph (HPLC) with UV visible detector (Perkin Elmer 200 series)
- Gas Chromatograph with FID, TCD, ECD, FPD Detectors (Perkin Elmer Clarus 500)
- Thermo-Gravimetric and Differential Thermal Analyzer (TG/DTA) (Diamond Series TG/DTA Perkin Elmer , USA), Max. Temperature 1300°C
- Dynamic mechanical Analyzer (DMA) (Perkin Elmer DMA, USA) for mechanical testing (at -165°C to 800°C)
- Differential Scanning Calorimeter (DSC) (Perkin Elmer Diamond Series DSC, USA), Temperature Range -65 to 600°C
- Surface Area Analyzer (NOVA 2200e Quantachrome, USA) using N₂ as purge Gas
- Ion Beam Thinner (Gatan PIP491, USA) for TEM samples
- Polishing Machine (Benetec, UK)
- Cutting Machine (Benetec, UK)
- Distilled water unit
- Sputter Coater and Vacuum Evaporator for SEM and TEM sample preparation
- Electrical Furnace (Nabertherm, Germany) 1200°C
- 200KVA Diesel Electric Power Generator (Broad Crown, UK)

All these equipments are the latest of their kind and highly useful for material characterization. Now the CRL has been
supplemented by an attached Materials Research Laboratory (MRL) within the Physics Department with facilities for processing of test materials and fabrication of test samples. Other property measurement instruments are in the pipeline to add state of the art property measurement kits to the existing facilities. Within the adjacent science departments and centres including chemistry, biotechnology, environmental sciences, pharmacy and others have established laboratories for relevant materials processing, formulation and fabrication, and further up-gradation work is in progress within these departments and centres for further facilitating their abilities of processing and fabrication of materials related to their disciplinary sectors. These will further enhance the utilization of CRL. The objective of these additional facilities is to facilitate the formulation and specification guidelines and standards for innovations in materials of industrial significance. To sum up the CRL as well as the collateral materials processing and sample fabrication facilities at the nearby premises no doubt enhances the capability and role of CRL to be unique of its kind throughout the country. It is felt necessary to describe the CRL equipments with some details so as to help new researchers become familiar regarding their uses.

Electron Microscopes

Electron Microscopes (EM) were invented to cope with the limitation of Light Microscopes in their ability of fine resolution (<0.2nm) and magnification. The theory involved behind the EM concept appeared in the 1930s with the consequent invention of EMs. They basically belong to the category of microscopes that use a beam of high energetic electrons instead of light photons for ultra-fine scale examination of material samples. Such examinations with conventional EMs enable analysis of surface features at sub-micron level depicting morphological and structural characteristics. It also enables the analysis of defects, impurities and extent of micro-structural homogeneity and phases. Additionally, the elemental composition of the samples under-examination can be investigated by an attached Energy Dispersive X-ray (EDX) detector. For further details of the operations and related EM techniques the interested readers may consult references 1-5.

The Transmission Electron Microscope (TEM) was the first type of Electron Microscope developed in which a focused beam of electrons controlled by electromagnetic lenses were used to see through electron-transparent specimens. Knoll and Ruska in Berlin Technische Hochschule (Germany) developed TEM in 1931 whereas Siemens Company of Germany produced the first TEM for commercial use. Similarly, the idea behind the development of Scanning Electron Microscope (SEM) in 1932 was materialized as commercially available instrument in 19651-6.

In all electron microscopes, a stream of electrons is produced by the electron source (electron gun). The produced electrons are accelerated towards the specimen using a positive electrical potential. This stream is focused by metal apertures and magnetic lenses into a thin, focused, monochromatic beam. The resulting interactions occurring between the irradiated sample and electron beam produce new signals with their intensity dependent upon the sample characteristics. The interactions and effects are translated onto a fluorescent screen as an image by detectors. These interactions are produced in all electron microscopes regardless of their type. There are two main types of the electron microscopes1-7.

- Scanning Electron Microscopes
- Transmission Electron Microscope

Transmission Electron Microscope (TEM)

The Transmission Electron Microscopes (TEMs) Shown in Fig.1, are patterned after Transmission Optical Microscopes. In the case of TEM, a focused electron beam falls on a very thin electron transparent sample instead of the light radiations as in the Transmission Optical Microscopes. The information given is similar to that of Transmission Optical Microscopes, but has very high magnification and resolution2-5. The size, shape and distribution of crystals in the specimen, and morphology and extent of crystal defects to sub-micron level can be studied using TEMs2-5. Additionally, the use of High and Ultra Resolution Transmission Electron Microscopes (UHTEM) facilitates studies up to atomic level (of the order Å).

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Scanning Electron Microscope (SEM)
Scanning electron microscopes (SEM), shown in Fig. 1b, are patterned on the principle of the Reflecting Light Microscopes. In SEMs, the incident beam of electrons interact with the sample and produce signals like secondary electrons, back scattered electrons, X-rays, Auger electrons and cathodoluminescence which can be used for examination of surface features and chemical analysis of a test sample\(^1\). SEM is generally used to investigate the gross microstructure of the specimen such as grain, phase and size distribution of constituents. The Energy dispersive X-ray spectroscopy (EDX) system attached to SEM can detect elements ranging from boron to uranium along with their relative atomic and weight percentages in microscale area of interest\(^2\). EDX is a type of spectroscopy that allows elemental detection by using the fundamental principle that each element of the periodic table has a unique electronic structure and thus emits a unique X-ray spectrum upon interaction with high energy electrons with material atoms\(^1\). This results in the emission of a specific amount of energy, which is characteristic for that specific element. No two elements in periodic table have the same values of energy in K, L or M shells. The energy values of the characteristic X-rays generated from sample are measured by a detection system which are plotted against intensity, and thus atomic and weight percent of the constituent element are determined\(^1,5\).

X-ray Diffractometer (XRD)
The X-rays are electromagnetic radiations of wavelength shorter than ultraviolet radiations and longer than \(\gamma\)-rays. These are high-energy photons with wavelengths of the order of 1 Å and are thus comparable with atomic dimensions\(^5,7,9\).

Diffraction occurs when electromagnetic radiation (X-rays) impinges on periodic structures with geometrical variations on the length scale of the order of the wavelength of the incident radiation. The inter-atomic distances in crystals and molecules are of the order 1.5–4 Å which correspond to the photon energies ranging from 3 to 8 keV. Accordingly, phenomena like constructive and destructive interference become observable when periodic arrays of atomic planes are exposed to X-rays. According to Bragg’s law \((n\lambda = 2d\sin \theta)\), the rays will interfere constructively when the path difference of the two rays is an integral multiple of the wavelength of the radiation\(^2,5,8-9\).

The XRD technique using the powder method is commonly used for crystallographic analysis. In this method, the use of finely powdered specimens ensures the random orientation of crystalline planes to satisfy the Bragg’s diffraction conditions. Current generation XRD machines (Figure 2) can detect the angle and the relevant intensity of the diffracted beam with an inherent radiation counter. A recorder attached to the system automatically plots the intensity of the diffracted beam according to the motion of the counter on goniometer circle. The diffraction angles of the beams and their respective intensities can be recorded at the same time by this method of the crystal analysis\(^2,5,8-10\).

Universal Testing Machine (UTM)
Universal Testing Machines (UTM) are suitable for studying the behaviour and mechanical properties of solid materials and objects made from these. In UTM, test specimens are subjected to loadings causing their stretching, bending, compressing, or pulling at a controlled speed until they break. UTM is the best device for testing of bench properties of any material ranging from biomaterials to metals and alloys.

In UTM the load cell is attached to a moving crosshead that is driven by electric motors or hydraulics and measures the force. UTMs have interchangeable load cells for different...
ranges of the material being tested. UTMs apply force to a specimen in order to induce strain in the material. The particular test (tensile, flexural, compression or shear) is classified in terms of the direction of introducing the strain in a specimen and the speed of the applied force. Unlike their earlier versions, modern UTMs are equipped with automatic control and digital recording and printing systems. A test is thus preset to run automatically with the facility for display of the data at a test progression stage. A printout of data or its plotting is obtained through an inbuilt computer system without the need for manual calculations and plotting.

Thermal Systems
The thermal analysis techniques facilitate the measurement of the variation in a physical property of a material in response to changes in temperature. The thermal behaviour of a material thus studied can be used as guideline for setting standards for product development and designing. The widely used methods for the thermal analysis include:
- Thermogravimetry (TG)
- Differential thermal analysis (DTA)
- Differential scanning calorimetry (DSC)
- Dynamic mechanical analyzer (DMA)

Thermogravimetry (TG)
In TG analysis, the mass of a sample in a controlled atmosphere is recorded continuously as a function of temperature or time as the temperature of the sample is increased. The TG provides the information about the decomposition, oxidation reactions and the physical processes like vaporization, sublimation, and desorption. The most important applications of TG are the study of polymers, which provides information about the mechanisms of decomposition for various polymeric arrangements. As the decomposition patterns are characteristic for each polymer, it is utilized for the identification of the polymer materials.

Differential Thermal Analyzer (DTA)
In DTA (Figure 3a), the difference in the temperature of sample and a reference material is measured as a function of temperature while the substance and reference material are subjected to a controlled temperature program. Usually, the temperature program involves heating the sample and reference material simultaneously. The test sample undergoes phase transformation or chemical change with temperature while the reference material remains unchanged in that temperature range. In this way, the heat absorption or emission from the test sample is recorded in terms of ∆T which is plotted against sample temperature, thus giving peaks or dips when significant phase transformation events occur. DTA is utilized for the determination of thermal behavior during the increasing temperature and composition of naturally occurring and manufactured products. Fusion, vaporization, sublimation, absorption, desorption, dehydration, reduction and decomposition are the endothermic physical processes. Adsorption, crystallization, oxidation, polymerization, and catalytic reactions are the exothermic processes.

Differential Scanning Calorimetry (DSC)
DSC (Figure 3b) is also a thermal technique in which differences in heat flow into a sample and a reference material are measured as a function of sample temperature while the two are subjected to a controlled temperature program. The basic difference between differential scanning calorimetry and differential thermal analysis is that in DSC the differences in energy are measured while in DTA differences in temperature are measured. The temperature programs for the two methods are similar. DSC is used to characterize the crystallization behavior of the materials. DSC has found extensive use in the pharmaceutical industry for testing the purity of drug samples.

Dynamic Mechanical Analyzer (DMA)
Dynamic Mechanical Analysis (DMA) shown in Figure 3c and Dynamic Mechanical Thermal Analysis (DMTA) or Dynamic Thermomechanical Analysis are utilized for characterization of the thermomechanical properties of materials in the changing temperature environment. The viscoelastic nature of polymers can be studied by DMA. Currently there are two methods used for DMA. One involves the decay of free oscillations and the other forced oscillations. In the free oscillation technique, upon removal of an applied force, sample oscillates. In contrast, in the forced oscillation method, a sample is kept underload and simultaneously subjected to an oscillating force and the resultant displacement of the sample is measured. The most common method used today is the forced oscillation method. Linear strains, bending, tension, shear and compression are applied to test solid samples. Under the applied load sample deforms, and stiffness of the sample is determined. The damping properties (tanδ) of the material can be
determined by measuring the time lag in the
displacement compared to the applied force\textsuperscript{12-13}.  

Figure 3a: TG/DTA  

Figure 3b: DSC  

Figure 3c: DMA  

Chromatography  
Chromatography is an important group of methods that allow the separation of closely associated components of complex mixtures. In all chromatographic separations, the sample is transported through a mobile phase (a gas or a liquid). This mobile phase is then forced through a non-homogeneous stationary phase, which is fixed in a column or on a solid surface. The two phases are chosen so that the components of the sample are distributed between the mobile and stationary phase at varying degrees. Those components that are strongly held by the stationary phase move slowly with the flow of the mobile phase, while the components that are weakly held by the stationary phase travel rapidly. As a consequence of these differences in mobility, sample components separate into discrete bands or zones that can be analyzed both qualitatively and quantitatively\textsuperscript{8}. Among the different chromatography techniques, the most common include Gas Chromatography (GC) and High Performance Liquid Chromatography (HPLC).

Gas Chromatography (GC)  
In GC, a gaseous or a volatile sample is vaporized and injected into the chromatographic column. Separation is produced by the flow of an inert gaseous mobile phase. The mobile phase has no interaction with molecules of the analyte. It only transports it through the column\textsuperscript{8}. GC is used for the determination of the purity of organic compounds and identification and separation of components of complex mixtures.

High Performance liquid Chromatography (HPLC)  
In HPLC, the analyte is forced through a column of the stationary phase by pumping a liquid mobile phase at high pressure through the column. A small volume of the sample to be analyzed is introduced to the stream of the mobile phase. The sample is retarded by specific chemical or physical interactions with the stationary phase as it flows through the column\textsuperscript{8}. The HPLC is utilized for the separation of DNA from different sources, measurement of catecholamines and metabolites, and for determination of the purity of medicines and polymers\textsuperscript{8}.

Atomic Absorption Spectrometry (AAS)  
Atomic absorption spectrometry (AAS) is an analytical technique used for the measurement of elemental concentrations in a liquid sample. Also the known mass of a solid sample can be dissolved in a specific solvent facilitating the compositional analysis of the dissolved material. Atomic absorption is a sensitive technique and can measure the very small concentration of an element in parts per million (ppm) or part per billion (ppb) in a sample. In this technique, use is made of an emitted wavelength of light by a lamp and its absorption by an element present in the sample, which corresponds to the energies required for transporting electrons from lower energy levels to higher energy levels\textsuperscript{8}. Atomic absorption spectrometry has many uses including analyzing metals in biological fluids, finding out the levels of various elements in water, air and petrol.
identification of toxic impurities, and for the determination of amount of metals such as gold or other precious metals in rocks. 

**Surface Area Analyzer**

Two important physical parameters that determine quality and utility of materials are the surface area and porosity. Variations in the particles surface area and porosity of material greatly influence its performance. For the determination of surface area, surface area analyzers and pore size analyzers are used. The samples treated by combination of heat, vacuum and/or flowing gas to remove adsorbed contaminants acquired because of its exposure to atmosphere, and then it is cooled, under vacuum conditions. An adsorbent, usually nitrogen, is admitted to the solid sample in controlled increments. On equilibrium of pressure after every dose of adsorbent in the surface area or pore size analyzer, the adsorbed quantity of gas is calculated. The adsorbed volume of gas at each pressure describes an adsorption isotherm, from which the quantity of gas required to form a monolayer over the external surface of the solid particles and its pores is determined. By using the area covered by adsorbed gas molecule, the surface area of the particles of the sample can be calculated. Surface area analyzers are also used for the determination of pore distribution, pore volume, specific surface area and total surface area of the materials.

**Utilization of the CRL**

Since its inception, the facilities and equipments at the CRL are regularly used by researchers and students of many institutes and centres within the UOP campus and NWFP University of Engineering and Technology, Peshawar, National Centers of Excellence in Physical Chemistry and Geology, Department of Chemistry, Botany, Pharmacy, Biotechnology and Physics. Other national level institutes and centres are also users of the facilities at the CRL. Among these are included: National Institute of Biotechnology and Genetic Engineering, (NIBGE) Faisalabad, Quaid-i-Azam University Islamabad, Gulham Ishaq Khan Institute of Engineering Sciences and Technology, Topi, Swabi, Pakistan Institute of Applied Sciences and Engineering, (PIASE) Islamabad, Bahauddin Zakariya University Multan, National Centre of Excellence in Molecular Biology, The University of Punjab, Lahore. The facilities are also extended for use by private institutions and industries. Before the installation of these equipments, the test samples had either to be sent to overseas laboratories or to those located at faraway places, if any within the country. These were ordinarily inaccessible, expensive to use and frustrating to the researchers because of prolonged delays in acquiring the test results.

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**REFERENCES**