Equipment - Key Centre for Polymers and Colloids

The Key Centre for Polymers and Colloids hosts a unique facility for the preparation and characterisation of polymers, colloids, and surfaces alike. Our equipment suite includes traditional and specialist organic synthetic apparatus, instrumentation for the characterisation of polymers, colloids, and for generic surface chemistry. We also have specialised apparatus for the synthesis of polymer colloids, including the Mettler RC1 and custom-built instruments such as dilatometers. The KCPC also has access to the pool of instruments available within the University, with privileged links with the School of Chemistry and The Australian Centre for Microscopy & Microanalysis (ACMM).

Material Characterisation

Molecular Weight and Particle Size Analysis

Size Exclusion Chromatography (SEC)



Shimadzu SEC instrument

SEC is commonly used in our laboratories to determine the molecular weight and molecular weight distribution of the polymeric materials prepared. We currently have a UFLC Shimadzu LC-20AD prominence liquid chromatograph running N,N-dimethylacetamide at a flow rate of 1 mL min⁻¹ at 50 °C. This system can accurately quantify the molecular weight of polymeric samples ranging between 3,000 to 1,500,000 g mol⁻¹.

Specifications:

- SIL-20A HT prominence auto sampler
- DGU-20A₃ prominence degasser
- Rid-10a Shimadzu RI detector
- miniDAWN TREOS Wyatt LS detector
- Wyatt ViscoStar-II viscometer

Light Scattering (LS)

Light scattering is used in the KCPC primarily to assess the particle size of materials such as iron oxide and silica nanoparticles before and after modification, as well as to investigate the self-assembly of multiblock copolymers under various conditions. The KCPC currently houses a number of instruments including a Malvern Mastersizer 2000, and a Malvern Zetasizer Nano ZS.

Malvern Mastersizer 2000



Malvern Mastersizer 2000

The Mastersizer 2000 particle size analyser can measure the size and size distribution of spherical particles with diameters ranging between 20 nm and 2000 μ m.

Specifications:

- He-Ne laser (red light)
- Solid-state laser (blue light)
- Accurate within ±1 %
- Highly reproducible

Malvern Zetasizer Nano ZS (DLS)



Malvern Zetasizer

The Zetasizer is ideal for measuring the particle size and distribution of spherical particles as small as 0.3 nm, the electrophoretic mobility of proteins, the molecular weight of macromolecules, and the zeta potential of colloids and nanoparticles alike.

Specifications:

- He-Ne laser (633 nm, 4 mW)
- 173° fixed scattering angle
- Temperature control between 0 and 90°
- Small sample volumes required

- 0.3 nm-10 μm diameter range
- Zeta potential measurements
- Molecular weight measurements (980 Da–20 MDa)
- Malvern MPT-2 Multi Purpose Titrator accessory, allowing the zeta potential, size or intensity measurements to be made whilst adding a titrant to the sample to investigate the effect of pH, dilution or an additive

Hydrodynamic Chromatography (HDC)



PL-PSDA

Hydrodynamic chromatography is used in the KCPC to measure the size distribution of negatively charged particles that are suspended in a colloidal dispersion. HDC measurements are performed using our in-house Polymer Laboratories Particle Size Distribution Analyser (PL-PSDA). The PL-PSDA is equipped with a type 1 or type 2 column, which are packed with a bed of non-porous particles that separate particles based on size.

Specifications:

- Accommodates up to 87 samples with automated injection
- 1.5–2 mL of sample solution in eluent required at varying concentrations; lower particle concentrations for large particles, and higher particle concentrations for small particles
- PL-PSDA Control software for autoinjection and sample analysis
- Particle size distribution determined by peak fitting with a modified Pearson Type VII distribution model
- Size calibration performed with latex standards and marker
- Capable of separating particles in the 5–300 nm size range (type 1 cartridge), and the 20–1500 nm size range (type 2 cartridge)
- Suitable for samples that can be dispersed in water including latexes, emulsions, colloids, liposomes, and organic pigments

Optical Microscopy

Leica DM750 Optical Microscope



Leica DM750 Optical Microscope

Specifications:

- 4x, 10x, 20x, 40x, and 100x objectives
- Capable of brightfield, darkfield, phase contrast, and cross polarisation in transmitted light
- Leica ICC50HD high definition camera
- LAS 4.6 software package for the capture and editing of images

Mass Spectrometry (MS)

Mass Spectrometry is used predominantly to assess the molecular weight and dispersity of macromolecules, and to characterise the chemical structure and functionality of both small molecules and oligomers. The KCPC has access to a whole host of MS techniques (ESI, APCI, EI, CI, GC/MS, MALDI, high resolution) and instruments through the School of Chemistry Mass Spectrometry Unit.

Nuclear Magnetic Resonance (NMR)

Nuclear magnetic resonance is used to determine monomer conversion, assess the chemical structure, composition and chain length of polymeric species, as well as for small molecule characterisation. The KCPC has access to a Bruker AVANCE200, a Bruker AVANCE300 and Bruker AVANCE400 spectrometers through the Nuclear Magnetic Resonance Laboratory in the School of Chemistry at the University of Sydney. For more information about the instrumentation, please visit the website.

Surface Characterisation and Material Morphology

Spectrophotometry

The KCPC uses a variety of techniques including UV-Vis, FTIR and RAMAN that are available in the <u>School of Chemistry</u> and through the <u>Vibrational Spectroscopy</u> <u>Facility</u> at the University of Sydney.

Electron Microscopy

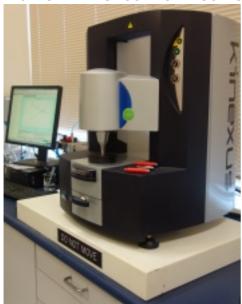
The KCPC uses a variety of electron microscope techniques including Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) in combination with spectroscopic techniques such as EELS and EDX to investigate the morphology and composition of various materials including polymer hybrids and blends to colloidal particles. For more information regarding these techniques and the instruments available, please visit the <u>Australian Centre for Microscopy & Microanalysis (ACMM)</u> website.

Atomic Force Microscopy (AFM)

Atomic Force Microscopy is used to investigate the surface roughness, phase separation and structure adopted in polymer blends. The <u>Neto Group</u> houses two AFM instruments in the School of Chemistry; the Digital Instruments Multimode 8, and the Asylum MFP-3D.

Rheometry

Malvern Kinexus Pro Rheometer



Malvern Kinexus Pro Rheometer

The Kinexus Pro is used to characterise the rheological properties of a range of materials from dispersions, polymers, as well as other complex fluids to soft solids. This is a powerful tool to capture and evaluate the changing viscoelastic properties of materials under various stress, strain and temperature conditions.

Operation Modes:

- Viscometry mode
- Oscillation mode
- Normal force mode

Accessories and Features:

- Parallel plates
- Cone and plate attachments of different diameters
- Active hood peltier plate cartridge, providing control over the environment to minimise thermal loss and therefore reduce thermal gradients
- 'Plug and play' functionality for all measuring systems and environmental control units
- Standard Operating Procedure-driven rheological testing
- Intelligent geometry recognition and auto-configuration
- Direct strain control, shear rate control and shear stress controlled studies
- Torque range from 2 nNm to 200 mNm with a resolution of 0.1 nNm
- Normal force range from 0.001 to 20 N with a resolution of 0.5 mN
- Fully configurable vertical profiles by speed and normal force
- Operating temperature between 15 and 40 °C

Thermal Properties

We use a variety of techniques to elucidate the thermal characteristics and behaviour of various polymeric materials prepared in our laboratories. Such techniques include Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) available through the Thermophysical Properties Facility in the School of Chemistry at the University of Sydney. The KCPC also performs hyperthermia testing using a nanoTherics magneTherm System.

nanoTherics magneTherm System



nanoTherics magneTherm System for hyperthermia testing

The nanoTherics magneTherm System is used to examine the heating capacity of magnetic materials.

Features:

- Large frequency range (100–1000 KHz)
- A wide sample aperture allows calorimetric, in vivo, in vitro and material experiments to be conducted
- · Vertical and horizontal sample positioning possible allowing nanoparticle samples and animal models to be examined

Specialised Reactors for Controlled Synthesis, Preparative Devices, and General Laboratory Equipment

Torrey Pines Scientific Echotherm IC20 Chilling/Heating Plate



Chilling/Heating Plate

The Echotherm dry bath is perfect for performing polymerisation kinetics for reaction optimisation, able to accommodate a maximum of 24 samples up to a temperature of $100\,^{\circ}\text{C}$. It is also suitable for cooling or heating biological samples for defined time periods. This includes incubation, enzyme/DNA library storage, sample transportation, PCR sample preparation, and performing ligation and hybridization reactions.

Features:

- Temperature control between -10 and 100 °C without the sample block, accurate to within 1 °C, and with a 0.3 °C uniformity
- The sample block can accommodate up to 24 samples
- Platinum RTD sensor
- PID controller
- 50 W power supply

Electrospinning



Spraybase

The Spraybase electrospinning system is used in the KCPC for electrospraying and electrospinning. It can be used to form colloids and nanofibres rapidly for various research needs.

This system has a variable high voltage power supply (up to 30 kV). A laser is used to illuminate the plume/Taylor cone for visualization. A computer controlled syringe pump module, which allows the user to control the flow rate through the emitter, is used. The system has a digital video camera connected to a computer to video the Taylor cone. The spray unit is housed in a transparent safety hood.

Features:

- Benchtop apparatus
- 30 kV controller
- Anti-vibration platform
- Spraybase Syringe pump module
- Camera for visualisation of Taylor cone characteristics
- Spraybase Emitters
- z-axis manual control between emitter and collector plate

Benchmark Incu-Shaker Mini



Benchmark Incu-Shaker Mini

Features:

- Compact shaking incubator, only 11" wide
- 300 W power supply
- Exceptional temperature uniformity within ± 0.25 °C
- Horizontal circular 19 mm orbit for aeration and mixing
- Variable speed of 30 to 300 rpm, with a 1 rpm increment
- \bullet Variable temperature between 5 and 70 °C, with an accuracy of ± 0.1 °C
- Compatible with a variety of platforms, flasks, and tubes
- Continuous timing from 1 min to 48 h

Centrifugation

We have a number of centrifuges available in our laboratory including a Beckman Coulter Optima XE-100 Ultracentrifuge, a Beckman Coulter Allegra X-30 Centrifuge, a Beckman Coulter Allegra X-22 Centrifuge, and a Benchtop Eppendorf Centrifuge.

Beckman Coulter Optima XE-100 Ultracentrifuge



XE-100 Ultracentrifuge

- Maximum speed of 100,000 rpm
- Aqueous samples
- Temperature control between 0 and 40 °C in 1 °C increments
- Accommodates up to 8 samples, with a maximum volume of 30 mL per sample

Beckman Coulter Allegra X-30 Centrifuge



X-30 and X-22 Centrifuge

- Maximum speed of 4,200 rpm
- Accommodates up to 16 samples, with a maximum volume of 50 mL per sample
- Compatible with 50 mL falcon tubes

Beckman Coulter Allegra X-22 Centrifuge

- Maximum speed of 14,500 rpm
- Accommodates up to 6 samples, with a maximum volume of 30 mL per sample

Benchtop Eppendorf Centrifuge

- Maximum speed of 14,500 rpm
- Accommodates up to 14 samples, with a maximum volume of 2 mL per sample
- Compatible with 1–2 mL Eppendorf tubes

Ultrasonication

Acoustic Box Ultrasonic Probe



Ultrasonic Probe

Sonication Baths



Branson Sonication Bath



Power Sonic410 Sonication Bath

Vortex Mixers



Vortex Mixer

Retsch S100 Ball Mill



Retsch \$100 Ball Mill

Specifications:

- 220 V
- 50 Hz
- 100 W
- 0.5 A

Other Equipment

- Avestin EmulsiFlex C50 High Pressure Homogeniser
- Ozonolyser
- Autoclave (Siltex Steam Sterilizer)
- VMA- Getzmann Dispermat Laboratory Disperser
- Brookfield Digital Viscometer

- Metrohm Karl Fischer Titration
- Pulsed Laser Polymerisation