

The BioComposites Centre

BioProducts and BioRefining -Technology Transfer Centre (BPR-TTC)



Overview

In 1989, The BioComposites Centre established an MDF (medium density fibre board) pilot plant at Bangor, North Wales with the backing of industry and the Welsh Development Agency. The aim of this facility was to provide technology transfer services and a research capability to the Forest Products Industry and other sectors. The facility carried out elements of what is now known as biorefining, by adding value to plant fibre. Feedstocks have included forest residues (including saw dust from mills and wood chips) and agricultural co-products such as wheat straw and hemp fibre, in order to produce biocomposite materials for a range of construction applications. Since 1989 a wide range of collaborative industrial projects have been undertaken at the facility to study the use of novel materials, new resins, additive technologies and process configurations.

The facility was relocated to its new site on Anglesey in 2006, with 615 m² of floor space. The remit of the BPR-TTC has since broadened to include work on the wider aspects of biorefining, including biomass fractionation and the study of a wide range of biobased products for application across many different sectors. These include the use of biopolymers such as polylactic acid and starch for food/ materials packaging, the isolation and modification of bioresins and other additives for the panel products/ polymers sectors and the extraction of speciality chemicals from biomass using supercritical carbon dioxide technology, for the cosmetics and nutraceuticals sectors.

The combination of a wide range of pilot scale equipment at the BPR-TTC, coupled with staff expertise in a number of core areas makes the facility unique and an ideal partner for working with industry. This document describes the facilities and summarises the technical specifications of the key components of the MDF facility and the other pilot scale equipment located within the BPR-TTC.

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TTC Equipment Capability



BPR-TTC capability



Pulp and Paper equipment



20 Litre rotary evaporator



Wet chemistry laboratory with walk in fume hood



50 Litre jacketed glass reactor



1000 litre drum blender

Pressurised refining of biomass



Office



Twin screw extruder and film forming line



Two deck vibrating sieve



Hot press for biocomposites production

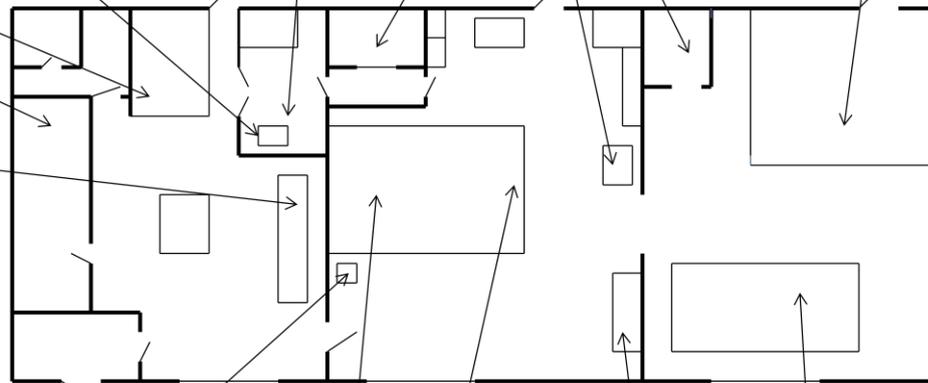


Pre press



Fibre collection and mattress forming station

Wet fractionation line – early 2013



1) Pressurised refining of biomass and biocomposites board production

The pilot plant MDF production facility at the BioComposites was designed and set up to replicate, on a much smaller scale, industrial MDF production. The only significant difference between industrial MDF production and the production at the BioComposites Centre is that in the former case hot pressing is by a continuous process. At the BioComposites Centre, the production process becomes a batch process at the press. The MDF production facility at the TTC is supported by ancillary sieving, hammer milling and drum blending equipment, used for batch fibre resination and mixing.

The facility's **ANDRITZ SPROUT-BAUER 12"** pressurised refiner consists of an in-feed hopper leading to an **MSD** (modular screw device, i.e. a plug feeder) which conveys the starting material (e.g. wood chips) from atmospheric pressure into the pressurised environment. Raw material is fed through the MSD and, via a 2.6 metre long cooker, to a 60-litre digester.



MSD (modular screw device)
and 2.6 m long cooker

60-litre digester

From the digester the material is fed by screw conveyor to the centre of a stationary refiner disc, and hence into the refining zone. The desired pressure is maintained in the system during the whole refining period by a constant supply of steam from a boiler. The residence time of material in the pressurised environment can be varied from 4 to 30 minutes. Steam pressure in the cooker/digester can be varied from 4 to 12 bar (gauge). The refiner plate gap and the refiner plates themselves can be changed in order to deliver varying levels of work to fibres.

The amount of work is recorded continuously allowing for the determination and comparison of energy used in the refining process. Fibre is vented from the refiner housing via a blow valve into a 9-metre long stainless steel **BLOWLINE**. Fibre resination takes place in the blowline. Additives such as wax (to reduce moisture absorption in the board) may also be incorporated at this stage. Addition rates for both wax and the resin are maintained by determining the oven dry fibre throughput, calculating the addition rate and then adjusting flow rates of appropriate pumps accordingly. Addition rates are monitored by computer.

The blowline is connected to a continuous, 120 metre long FLASH DRIER. The internal diameter of the dryer is 159 mm and the air for the dryer is heated via a hot oil heat exchanger (max 250 °C). Air velocity in the dryer is approximately 37 metres per second. These conditions give a total residence time for fibre in the dryer of 4–6 seconds. The dryer inlet temperature can be varied and energy usage can be determined. A cyclone system separates the dry fibre which can then be diverted directly to bags or to a pilot scale fibre collection and mattress forming station. The forming box is then transferred to the single daylight **PREPRESS** that has a platen area of 1.65 m by 1.65 m with a hydraulic self alignment system, panels are (cold) pre-pressed and then transferred to a hot press on caul plates. The daylight opening hot **MAIN PRESS** is capable of pressing boards 1 m by 1 m in dimension and is computer controlled using PressMAN software, which can be used to adjust panel thickness and other parameters. The software also monitors the programmed profile, internal gas pressure and core temperature of the boards produced. The platens are oil heated to a temperature of 120 °C-280 °C and are controlled by a hydraulic pump, allowing a maximum specific pressure of 45 kg/cm² to be achieved during the pressing process.



Fibre collection and mattress forming station



Pre-press



Hot-press

2) Pilot-scale chemistry processing unit



The pilot-scale chemistry processing unit at the TTC facility has recently been commissioned and is now fully operational.

The unit has a pilot-scale, bundled capacity of up to 50 litres for the extraction of bio-based feedstocks, synthetic reactions and mixing or blending of prototype products for BEACON partner companies or for contract research.

The key features are:

- External, PC-based operator control platform with shielded observation window;
- Intrinsically safe zone with CO₂ fire suppressant system;
- Temperature range: -120 °C to +200 °C by remote heater/chiller;
- Inert atmosphere if required;
- Multiple addition and condenser ports;
- Automated addition;
- Vacuum to 2 mbar by remote pump;
- Separate condenser and cold-finger chillers;
- Vacuum-assisted or peristaltic pump charging of solvents;
- Overhead stirrer with optional paddle configurations;
- Moderate over-pressure to facilitate discharge of products.

An adjacent wet chemistry laboratory facility allows work-up of biomass extraction and reaction products as well as exploratory small-scale biomass extractions and synthesis. This provides flexibility to work directly from the bench to pilot scale.

The key capacities are:

- Liquid-liquid separation up to 10 litres;
- Vacuum filtration up to 20 litres;
- Thin film rotary evaporation:
 - Bench top rotary evaporator;
 - Stand-alone rotary evaporator for 20 litre batch processing or semi-continuous operation and can be simply adapted for solvent reflux;
- Split-level fume cabinet with 304-stainless steel bund for small-scale procedures (up to 10 litres capacity);
- Pilot-scale chromatographic purification.

3) Labtech scientific twin screw extruder type LTE – 26 - 40 and LCR-300 Sheet/ film casting line



Diameter of screws:	26 mm
Mode of operation:	screws co-rotating
Screw rotation speed:	0-800 rpm
Motor power:	15 kw
Heating power:	500 W per cartridge
Maximum barrel temperature:	400 °C
Max. connecting amperage:	20A for 3-phase, 220V
Minimum water pressure:	2 bar
Water flow rate:	20 litres per minute at 2 bar

This equipment is used for the production of wood fibre and polymer composites and extrusion technology is used extensively for mixing, compounding, or creating polymeric materials. The 26 mm co-rotating twin screw extrusion equipment at the TTC incorporates a modular clam shell barrel design, with each module (barrel section) being 104 mm in length. The interchangeable segmented screws can be reconfigured to a specific application by selecting various kneading, mixing, and shear screw elements. The flexibility of twin screw extrusion equipment allows this operation to be designed specifically for the formulation being processed.

LCR-300 Sheet and film casting line



Suitable for producing films and sheet in many different resin types such as PE, PP, PS, ABS, PVC, PET, PC etc. and also biobased polymers.

- C-Clamp flange on flat film die for easy removal of chill roll attachment as well as changing of screens.
- Flat 200 mm wide coat hanger type film & sheet die with adjustable lips from 0 to 2.5 mm. For sheet thickness from 0.3 to 2.0 mm or film thickness down to 0.01 mm.
- Pressure sensor with incorporated melt temperature sensor inserted in the die adaptor near the breaker plate.

- Sheet and film chill roll attachment for either horizontal or vertical roll stack position.
- With large 145 mm diameter chill roll and additional smaller cooling and polishing rolls of 75 mm diameter. Roll widths are all 350 mm. All three rolls are equipped with internal cores for efficient water-cooling and heating, allowing a precise temperature control of roll surface.
- Swing aside control panel with:

Three digital RPM speed controls for controlling the speed of:

1. Chill rolls
2. Nip-Rolls for pulling out the film/sheet
3. Windup.

Three digital programmable self-tuning temperature controllers for the flat die.

Control buttons for:

1. Main selector switch for on/off of control circuit
2. Selector switch for on/off of high pressure blower to air knife
3. Selector switch for up/down movement of polishing roll
4. On and off buttons for Chill rolls, Nip rolls and Windup system.

- Hot and cold water circulating unit, connected to all 3 rolls and with temperature regulation for a maximum temperature of 140 °C
- Air knife with adjustable nozzle height and angle to the Chill roll, connected to a high-pressure ring blower.
- Output in range of 10-20 kg/h depends on resin type.

5) Liquid/ supercritical carbon dioxide extraction equipment

Extraction can be carried out using CO₂ in a liquid or supercritical state and the choice largely depends on the solubility of the molecules to be extracted. Liquid CO₂ is significantly less polar than supercritical CO₂ (scCO₂) and is applicable only to small, non-polar molecules. In the supercritical state CO₂ is a highly tuneable solvent and the manipulation of temperature and pressure allows selective extract of a wide range of molecules. Supercritical CO₂ has low surface tension and viscosity and therefore high mass transfer rates can be achieved, providing ideal conditions for extracting compounds with a high degree of recovery in a short period of time and with easy separation of products.

Some of the applications already developed using CO₂ as a solvent include:

- Extraction of herbs and spices for food and beverage use
- Extraction of waxes and oils for cosmetic, personal care products and nutraceuticals
- Extraction and fractionation of pharmaceutical molecules
- Recovery of valuable molecules from end-of-life electronics
- Synthesis of flavour and aroma molecules using biocatalysts to meet new EU legislation

The main benefits of carbon dioxide extraction technology include:

- More efficient and environmentally friendly than traditional organic solvent based extraction techniques, with comparable or lower operating costs and energy requirements
- Highly selective isolation of different classes of plant chemicals by varying temperature and pressure, leading to pure, stable extracts with high levels of bioactive molecules
- Low temperature and pressures generally used for processing ensure that neither the extracts or residual material are degraded during the procedure
- Solvent free process with the potential to recycle the solvent for continued reuse
- Extracts produced using this technology are safe for use in both food and medicinal applications

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The equipment within the TTC allows trials to be carried out on as little as 5 g of material and is supported by excellent pre-treatment facilities and analysis of raw materials and products. This additional flexibility means that small scale extraction protocols can be optimised at 10 ml or 100 ml scale, prior to scale up using the 1 litre extraction vessel.

To support commercial development of new products based on scCO₂ extracts it is often necessary to provide kilogram quantities of materials to enable formulation trials to be carried out. Access to full commercial scale (>500 litre) plants in Europe is not a problem and the TTC works closely with large scale extraction companies to provide an opportunity to produce commercial quantities. However, there is no intermediate pilot plant available in the UK at all and very few available at sites in Europe. The availability of a pilot scale (up to 30 litres) extraction plant at Bangor capable of operating at similar pressures and temperatures to the existing laboratory plant is highly desirable in order to provide the bridge between laboratory and full commercial scale extraction. Provision of a capital grant from the Welsh Government has allowed further investment to purchase this type of equipment, which was commissioned in August 2012.

Small Scale Extraction Equipment



Pilot Scale Extraction Equipment



Equipment Specifications

Extraction capacity: 10 ml, 100 ml and 1000 ml

Operating pressure range: 50 - 600 bar

CO₂ Flow Rate: Max. 200 g/min

Operating temp: up to 120°C

Separator capacity: 25 ml or 500 ml

Solvent: Liquid or supercritical carbon dioxide with ancillary pump for high pressure addition of co-solvents

Extraction capacity: 2 x15 litres (maximum) or 2x 12 litres with extraction baskets

Extraction operating pressure: up to 700 bar

CO₂ flow rate: 10 to 50 kg/h

Temperature ranges: 5 to 80°C (for main autoclaves)

Separator capacity: 2 x 1 litre

Solvent: Liquid or supercritical carbon dioxide with ancillary pump for high pressure addition of co-solvents

6) High intensity, large scale sonic processing cell



Ultrasonication has been applied widely in various biological and chemical processes and has considerable value as a pre treatment step for biomass, resulting in disruption of the plant cell structure prior to downstream extraction or additional processing of the material.

The equipment at the BRP-TTC consists of a small scale ultrasonic probe suitable for preliminary experiments at 2 litre volume. The process can be scaled up using a 37 litre ultrasonic cell.

The system comprises of:-

i. Processing Cell

- one 37 litre stainless steel (AISI 316) cell with covers.
- Internal dimensions- 313 mm diameter x 480 mm.
- Internal wall polished and hard chrome plate.
- Transducer system consisting of a prestressed piezo electric sandwich.

ii. Generator

- Six solid state power amplifiers each having a power output of 600 watts average, 1200 watts peak.
- Six generators housed in one 9U 19 inch rack mounted cabinet.
- An automatic frequency control system maintains optimum tuning.

7) Wet biomass fractionation line

This will be commissioned by early 2013. More details to follow

8) Pulp and paper equipment

The equipment at the TTC provides independent technical support to industry and offers a wide range of testing services. Equipment located in the TTC includes:

Sprout Waldron 12" atmospheric refiner.

Disintegrators: With revolution counter.

Screening: Somerville screen for the removal of shives.

Bauer-Mcnett Pulp Classifier: Routine determination of fibre length distributions of all types of pulp samples.

Freeness Testing: Freeness (or drainage) of pulp samples. Canadian Standard freeness and Schopper Riegler testers are available.

Kajaani FS-200: To determine fibre length using automated method by which the numerical and weighted average fibre lengths and fibre length distributions of pulp can be measured.

Handsheet Production and Testing: Semiautomatic handsheet former. Burst tester. Brightness. Tear tester. Automated Micrometer.

Deinking Equipment: Laboratory scale Lamort pulper with deinking facilities.

9) Analytical capability

In addition to the wide range of processing capability that the TTC has, the work is supported by state-of-the art analytical techniques. The department has GC, GC-MS, ion chromatography, analytical and preparative HPLC and as well as ICP for elemental analysis.

- Perkin-Elmer Clarus 680 GC-MS (EI and CI)
- Perkin-Elmer Autosystem XL GC with FID detection
- Varian Prostar analytical HPLC with DAD
- Varian Prepstar preparative HPLC with UV detector
- Dionex Ion Chromatograph