



ENFiber

1st ENFiber Training School

Fiber 101: Basics of Fibers
Workshop

PROTOCOL BOOK

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Separation techniques

FIBER 101 – DEMONSTRATION PROTOCOL

FROM PLANT EXTRACT TO COLOURED FIBRE: CURCUMINOID EXTRACTION, DYEING AND COLOUR MEASUREMENT

Topic: Separation techniques / Extraction and fibre dyeing

Instrument / technique: Radleys Carousel Reaction Station; COLORCATCH NANO 2 colorimeter (Colorix)

Sample: Curcuma longa (turmeric) powder and fibre/textile samples

Estimated demonstration time: ~30–40 min live demonstration

1. INTRODUCTION

What will we explore?

This demonstration follows a simple workflow from a natural raw material to a coloured fibre, with the main focus on showing the use of the Radleys Carousel Reaction Station for controlled extraction and dyeing. Curcuminoids are first extracted from turmeric using a selected solvent. A small amount of the resulting extract is then diluted with Milli-Q (MQ) water to prepare the dye bath, which is used to dye fibres at 40 °C for 1 h. After washing and drying, the colour of the dyed fibres is evaluated using a portable colorimeter.

Briefly explain:

- The process transfers naturally occurring coloured compounds from a plant matrix into a liquid extract and subsequently onto a fibre surface/material.
- It demonstrates how extraction conditions and solvent choice can influence the amount of colourant available for fibre dyeing and, consequently, the final colour obtained.
- The main outputs are a curcuminoid-rich extract and a visibly dyed fibre sample, followed by objective colour measurement of the dried fibre. The demonstration also illustrates how the same reaction platform can be used for controlled extraction and dyeing steps.

Learning objective

After this demonstration, you should be able to:

- understand the basic principles of solid–liquid extraction and fibre dyeing;
- identify key parameters influencing extraction and dyeing, including solvent choice, temperature, time and mixing;
- interpret the visible extraction/dyeing outcome and instrumental colour measurement of the final fibre;
- recognize how natural extracts can be applied to fibre-containing materials and how the resulting colour can be evaluated objectively.

2. THE PRINCIPLE

Solid–liquid extraction transfers soluble compounds from a solid plant matrix into a liquid solvent. Here, coloured curcuminoids are extracted from turmeric using controlled mixing and temperature in the Radleys Carousel system. The extract is then used to prepare a simple dye bath by mixing 0.1 mL of extract with 5 mL of Milli-Q (MQ) water. Fibres are exposed to this dye bath at 40 °C for 1 h, allowing the colourants to interact with the fibre material. After dyeing, the fibres are washed with water to remove excess or weakly retained colourant and are then dried. The final fibre colour is evaluated using a portable COLORCATCH NANO 2 colorimeter. In a full analytical experiment, UV–Vis measurements may also be performed on the dye bath before and after dyeing to follow changes in absorbance; however, UV–Vis analysis is not included in this demonstration.

Key concept:

Controlled heating and mixing enable reproducible extraction and dyeing conditions, while the visible and measured fibre colour provides a simple demonstration of the process outcome.

3. MATERIALS

Sample: *Curcuma longa* (turmeric) powder for extraction; fibre or small textile specimens for dyeing. Exact fibre composition, sample dimensions/mass and turmeric-to-solvent ratio: to be confirmed before the demonstration.

Equipment: Radleys Carousel Reaction Station for controlled heating and stirring; COLORCATCH NANO 2 colorimeter (Colorix) connected to a compatible smartphone/tablet.

Materials / chemicals: Turmeric (*Curcuma longa*) powder; extraction solvent; extraction solvent 2; fibre/textile samples; Milli-Q (MQ) water for preparation of the dye bath and washing.

Safety: Wear laboratory coat, safety glasses and suitable gloves. Handle extraction solvents according to their SDS and work in a fume hood where required. Take care around heated reaction vessels and stirring equipment. Curcuminoid extracts strongly stain skin, clothing and surfaces. Dispose of solvent-containing extracts and wash solutions according to laboratory waste procedures.

4. METHOD

1. Prepare the Radleys Carousel reaction station and suitable reaction vessels.
2. Add a defined amount of turmeric powder and extraction solvent.
3. Perform the extraction under controlled mixing and temperature. Exact extraction time and temperature will be fixed after the solvent system is selected.
4. Separate the solid turmeric residue from the liquid curcuminoid extract and visually compare the extracts, if two solvents are used.
5. Prepare the dye bath by mixing 0.1 mL of curcuminoid extract with 5 mL of Milli-Q (MQ) water.
6. Add the fibre/textile to the prepared dye bath and dye for 1 h at 40 °C with controlled mixing using the Mya/Carousel system.
7. During the live demonstration, show the setup and start/explain the dyeing step. Because participants will not wait for the full 1 h procedure, use pre-dyed samples to demonstrate the subsequent steps.
8. Remove the fibre and wash it with water to remove excess or weakly retained colourant.
9. Dry the washed fibre completely under consistent conditions.
10. Connect/calibrate the COLORCATCH NANO 2, place it consistently on the dried fibre surface and record the colour values. Perform replicate readings at representative positions where sample size allows.
11. Compare the untreated fibre with the dyed fibre(s).

What to observe: Operation of the Carousel station; formation and colour of the turmeric extract; preparation of the diluted dye bath; visible transfer of colour to the fibre; colour remaining after washing and drying; and the difference between untreated and dyed fibres measured with the portable colorimeter.

6. RESULTS

A visibly coloured curcuminoid extract is obtained from turmeric and is used to prepare the dye bath (0.1 mL extract + 5 mL MQ water). Fibres exposed to the dye bath are expected to develop a yellow/orange colour that remains visible after washing and drying. The demonstration primarily shows how the Mya/Carousel system can provide controlled conditions for the extraction and dyeing steps.

How do we interpret it?

The visible extract demonstrates transfer of coloured curcuminoids from turmeric into the extraction solvent, while the dyed fibre demonstrates subsequent transfer of colour from the prepared dye bath to the fibre. Colour remaining after washing provides a simple visual indication that part of the colourant is retained by the fibre. The COLORCATCH NANO 2 provides an objective way to compare the untreated and dyed samples. UV-Vis would normally provide additional information on the change in the dye bath before and after dyeing, but this analysis is outside the scope of the present demonstration.

Typical result / value: No fixed value is specified in advance. The main comparison is untreated vs. dyed fibre and the appearance and final colour of fibres prepared under otherwise identical conditions.

8. TAKE-HOME MESSAGE

1. The Mya/Carousel platform enables controlled heating and mixing for both extraction and fibre dyeing.
2. The dye bath is prepared by diluting 0.1 mL of curcuminoid extract with 5 mL of MQ water, and the fibre is dyed for 1 h at 40 °C.
3. After washing and drying, the resulting fibre colour can be assessed objectively using a portable colorimeter.

WANT TO KNOW MORE?

Instrument / technique:

- Radleys Mya 4 Reaction Station: <https://www.radleys.com/range/mya-4-reaction-station/>
- Radleys Carousel systems: <https://www.radleys.com/>
- COLORCATCH NANO 2 (Colorix): <https://www.colorix.com/en/products/colorcatch-nano-2>

Viscoelasticity

FIBER 101 – DEMONSTRATION PROTOCOL

Gelation monitoring and evaluation of viscoelastic properties of hydrogels

Topic: Viscoelasticity

Instrument / technique: Rheometer MCR 303, Anton Paar

Sample: Alginate/CaSO₄ hydrogel

Estimated demonstration time: 20 min + hands-on experiments

1. INTRODUCTION

What will we explore?

Hydrogels are soft materials that combine liquid-like and solid-like behavior. During this demonstration, we will observe how an alginate solution rapidly transforms into a viscoelastic hydrogel after mixing with calcium sulfate (CaSO₄). Using an Anton Paar rheometer, we will monitor the gelation process in real time and determine the mechanical properties of the formed gel.

This technique is widely used for the development of biomaterials, bioinks, wound dressings, and fiber-containing hydrogel systems because it provides information about gel formation, stiffness, and structural stability.

Learning objective

After this demonstration, you should be able to:

- understand the basic principle of rheological measurements;
- explain the difference between elastic and viscous behavior;
- identify the gel point during hydrogel formation;
- interpret storage modulus (G') and loss modulus (G'');
- recognize how rheology can be used to optimize hydrogel-based fiber materials and biomaterials.

2. THE PRINCIPLE

Rheology studies how materials deform and flow when subjected to an external force. In oscillatory rheological measurements, a small sinusoidal deformation is applied to the sample and its response is recorded.

Two important parameters are measured:

- Storage modulus (G') represents the elastic, solid-like component and indicates how much energy is stored during deformation.
- Loss modulus (G'') represents the viscous, liquid-like component and indicates how much energy is dissipated.

When alginate is mixed with calcium ions released from CaSO₄, ionic crosslinks are formed between alginate chains. As crosslinking progresses, the material transforms from a predominantly liquid state ($G'' > G'$) to a gel state ($G' > G''$). Monitoring this transition allows direct determination of gelation kinetics and final gel strength.

Key concept:

A hydrogel is considered formed when the elastic response (G') becomes greater than the viscous response (G''), indicating the development of a stable three-dimensional network.

Scheme:

- Mix alginate solution and CaSO_4 .
- Start timer.
- Transfer the mixture onto the lower measuring plate.

Step 3.

- Lower the upper plate to the defined measuring gap.
- Remove excess sample around the plate edge.
- Begin measurement.

Step 4.

- Run the Experiment.
- Perform a time sweep while continuously monitoring:
 - Storage modulus (G')
 - Loss modulus (G'')
 - Complex viscosity

Step 5.

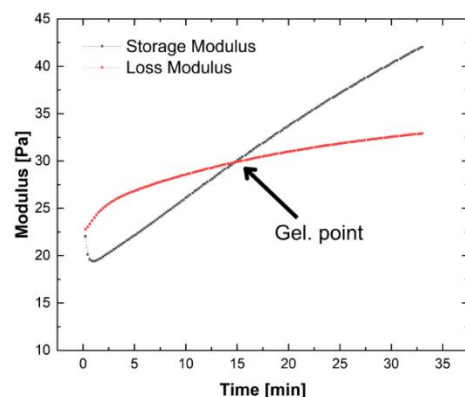
- Analyze Results
 - Identify the crossover point where G' exceeds G'' .
 - Determine gelation time.
 - Evaluate final viscoelastic properties.
- What to observe
 - Initially low modulus values.
 - Rapid increase of G' during crosslinking.
 - Transition from liquid-like to solid-like behavior.
 - Stabilization of G' after gel formation.

5. RESULTS

Expected result:

Immediately after mixing, both G' and G'' are low and the sample behaves mainly as a viscous liquid.

As calcium ions diffuse through the alginate solution, crosslinking occurs and G' increases rapidly. Within approximately 5 minutes, G' becomes higher than G'' , indicating successful gel formation. The storage modulus continues increasing until reaching a plateau corresponding to the final hydrogel stiffness.



How do we interpret it?

The increasing storage modulus demonstrates the formation of an interconnected calcium-alginate network. The moment at which G' exceeds G'' is often considered the onset of gel formation. Higher final G' values indicate a stronger and more elastic hydrogel structure.

The gelation rate and final stiffness can be adjusted through alginate concentration, calcium concentration, temperature, and mixing conditions.

Typical result:

Gelation time: ~10 min

Final G' : 0,1–10 kPa (depending on formulation)

Final $G' > G''$ indicating stable hydrogel formation

6. TAKE-HOME MESSAGE

Things to remember

1. Rheology measures how materials deform and flow under applied forces.
2. The relationship between G' and G'' reveals when a liquid transforms into a gel.
3. Rheological measurements are essential for optimizing hydrogel processing, fiber reinforcement, biofabrication, and biomaterial performance.

Rheology helps us to monitor hydrogel formation in real time and quantify the mechanical properties of soft materials.

WANT TO KNOW MORE?

Instrument:

Anton Paar Rheometry Portal:

<https://www.anton-paar.com/corpen/products/details/modular-compact-rheometer/>

Anton Paar d.o.o.

Tbilisijska 57 b

1000 Ljubljana

Slovenia

Email: info.si@anton-paar.com

T: +386 (0)1 423 4005

www.anton-paar.com

Further reading:

1. Škopek, Vojtěch, Ewa Olewnik-Kruszkowska, Magdalena Gierszewska, et al. 2027. "Alkaline Resistant Tunable Superaerophobic Bio-Based Chitosan/Dialdehyde Starch Hydrogels." *Biomass and Bioenergy* 217 (February): 109799. <https://doi.org/10.1016/j.biombioe.2026.109799>.

Registry Court: District Court in Ljubljana, 7189192000 VAT ID No.: SI93321490

Great people | Great instruments

2. Čič, Maja, Nejc Petek, Iztok Dogša, et al. 2025. "Sustainable Cyclodextrin Modification and Alginate Incorporation: Viscoelastic Properties, Release Behavior, and Morphology in Bulk and Microbead Hydrogel Systems." *Gels* 11 (11): 875. <https://doi.org/10.3390/gels11110875>.
3. Osojnik Črnivec, Ilja Gasan, Tigran Neresyan, Yuliana Gatina, et al. 2021. "Polysaccharide Hydrogels for the Protection of Dairy-Related Microorganisms in Adverse Environmental Conditions." *Molecules* 26 (24): 7484. <https://doi.org/10.3390/molecules26247484>.

Hydrogel preparation

FIBER 101 – DEMONSTRATION PROTOCOL

PREPARATION OF CALCIUM ALGINATE HYDROGEL BEADS BY PRILLING BY VIBRATION

Topic: Hydrogel preparation

Instrument / technique: BUCHI Encapsulator B-390

Sample: 2% (w/v) sodium alginate in deionized water, coloured with a small amount of water-soluble food colouring

Estimated demonstration time: 45-60 min (approximately 15-20 min live demonstration + 30 min hardening)

1. INTRODUCTION

What will we explore?

This demonstration shows how a liquid biopolymer solution can be transformed into hydrogel beads. A coloured sodium alginate solution is delivered through a single nozzle, broken into uniform droplets by controlled vibration, and collected in a calcium chloride hardening bath. The droplets gel as calcium ions crosslink the alginate chains, producing visible calcium-alginate hydrogel beads that can be handled and examined.

Learning objective

After this demonstration, you should be able to:

- understand the basic principle of ionotropic alginate gelation;
- identify how alginate concentration, calcium concentration, hardening time, and diffusion of Ca^{2+} affect the hydrogel bead;
- recognize characteristic hydrogel properties such as water retention, softness, shape retention, swelling, and permeability to small solutes;
- relate alginate hydrogels to immobilization, encapsulation, controlled release, and incorporation of fiber-derived or other functional materials...

2. THE PRINCIPLE

Sodium alginate is a hydrophilic polysaccharide containing negatively charged carboxylate groups. In water, its polymer chains are hydrated and mobile, so a 2% alginate preparation behaves as a viscous solution. When droplets enter the CaCl_2 bath, Ca^{2+} ions diffuse into the alginate and coordinate between neighbouring chains. These ionic junctions connect the chains into a three-dimensional network often described by the simplified “egg-box” model. Water remains inside the network, so the product is a hydrogel rather than a dry polymer particle. Gelation begins rapidly at the bead surface and continues as Ca^{2+} penetrates inward.

The B-390 contributes primarily by controlling geometry. It forms a stable liquid jet and breaks it into droplets that enter the calcium bath. Vibration, electrostatic dispersion, and stroboscopic observation are useful for producing separated, similarly sized droplets, but the key material transformation occurs only when alginate meets Ca^{2+} .

Key concept:

Controlled droplet formation determines bead size and uniformity, while Ca^{2+} -mediated crosslinking converts each alginate droplet into a hydrogel bead.

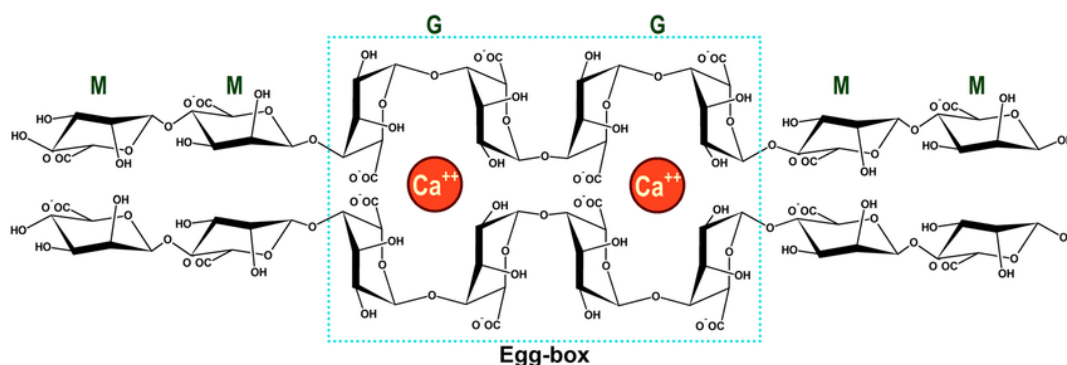


Figure 1. Schematic of "Egg-box" structure in alginate hydrogel crosslinked with Ca^{2+} ¹

3. MATERIALS

Sample: 2% (w/v) sodium alginate, coloured with food colouring; prepare as a homogeneous, bubble-free polymer solution.

Equipment: BUCHI Encapsulator B-390 with single-nozzle bead-producing unit and 1000 μm nozzle; B-390 air-pressure delivery system with liquid-flow regulation valve; stroboscopic lamp; electrostatic dispersion system.

Materials / chemicals:

- Sodium alginate, 2% (w/v) in deionized water
- Water-soluble food colouring, small amount for visibility
- CaCl_2 hardening solution, 100 mM
- Deionized water for preparation and washing

Safety: This is an instructor-operated laboratory demonstration. Wear standard laboratory PPE. Alginate spills are slippery and food colouring can stain. The B-390 uses compressed air/pressure and an electrostatic dispersion system, so only trained staff should adjust the pressure, flow-regulation valve, electrode, or nozzle area. Follow the laboratory SOP and the B-390 operating manual for assembly, operation, shutdown, and cleaning.

4. METHOD

Short instructions, STEP-BY-STEP procedure:

1. **Preparing / loading:** Transfer the coloured alginate into the polymer-delivery mechanism. In the single-nozzle process, any material intended to be entrapped would normally be mixed into the polymer before this stage; here, no separate encapsulant is used.
2. **Pumping:** Start delivery of the alginate through the nozzle and establish a stable continuous liquid jet before applying vibration.
3. **Vibration generator:** Apply the instrument vibration using the validated setting for the installed nozzle and alginate solution.
4. **Droplet formation:** Observe how the continuous jet breaks into a regular chain of similarly sized droplets, one after another.
5. **Dispersion:** Under trained-operator control, engage the electrostatic dispersion function so the droplets repel one another and spread before reaching the hardening bath. This reduces droplet collision and coalescence.

¹ Lee, Hwa-Rim & Jung, Sang & Yoon, Sejeong & Park, Tae & Kim, Seongju & Shin, Hyun & Hwang, Dong & Jung, Sungjune. (2019). Immobilization of planktonic algal spores by inkjet printing. Scientific Reports. 9. 1-7. 10.1038/s41598-019-48776-z.

6. **Visualization:** Use the stroboscopic lamp positioned behind the droplet stream to make the jet breakup and droplet chain easy to see. The operator confirms that a stable, regular droplet pattern is present before collecting the beads.
7. **Formation / gelation:** Allow the droplets to fall into the gently agitated CaCl_2 bath. On contact with Ca^{2+} , the alginate crosslinks and the droplets are converted into calcium-alginate hydrogel beads.
8. **Collection and hardening:** Continue production for the desired demonstration volume. After the final droplet reaches the bath, allow the beads to harden for 30 min. Then collect them with a sieve or strainer and rinse thoroughly with deionized water.
9. **Demonstration endpoint:** Transfer a small portion of washed beads to a clean dish or beaker for participants to observe. The remaining particles may be used directly or subjected to further treatment, depending on the teaching activity.

What to observe:

- The change from a continuous alginate jet to an ordered droplet chain after vibration is applied.
- The spatial spreading of droplets before they reach the bath, which helps prevent droplets from merging.
- Rapid shape retention after the coloured droplets enter CaCl_2 , followed by progressive strengthening during the hardening period.
- Bead shape and size consistency: stable operation should give mostly spherical beads of similar size; tails, irregular shapes, or strongly variable bead sizes indicate unstable droplet formation.
- The contrast between the initial viscous alginate liquid and the final soft, water-rich hydrogel beads.

6. RESULTS

Expected result:

A batch of visibly coloured, soft, approximately spherical calcium-alginate hydrogel beads that retain their shape after removal from the CaCl_2 bath and washing.

How do we interpret it?

Uniform, spherical beads indicate that the liquid jet was broken into droplets reproducibly and that the droplets remained separated before gelation. The ability of the beads to retain their shape after washing demonstrates formation of a crosslinked hydrogel network. Irregular shapes, merged beads, or a broad range of bead sizes usually reflect instability in droplet production, air bubbles, unsuitable liquid flow, or a mismatch between the solution properties and the chosen nozzle/operating conditions.

8. TAKE-HOME MESSAGE

Three things to remember

1. Calcium ions crosslink sodium alginate and convert liquid droplets into water-rich hydrogel beads.
2. Reproducible beads depend on stable droplet formation; nozzle size, liquid flow, vibration, solution viscosity, and hardening conditions all matter.
3. Alginate hydrogels provide a simple biobased matrix that can be used to immobilize or carry fiber-derived particles, active compounds, cells, or other functional materials.

In one sentence:

Prilling by vibration helps us turn a biopolymer solution into uniform hydrogel beads with controllable size and useful matrix properties.

WANT TO KNOW MORE?

Instrument / technique: BUCHI Encapsulator B-390 - Prilling by Vibration, single-nozzle bead production

Further reading:

1. BUCHI Labortechnik AG. Encapsulator B-390/B-395 Pro Laboratory Guide. See Sections 2.3-2.4 (single-nozzle procedure) and Application Note 9.5 (2% alginate / Ca-alginate beads).
2. Whelehan, M.; Marison, I. W. (2011). Microencapsulation using vibrating technology. *Journal of Microencapsulation*, 28, 669-688.
3. Cuomo, S.; Rispo, F.; Coppola, B.; Bedini, E.; D'Agostino, M.; Cassese, E.; Palmero, P.; Schiraldi, C.; La Gatta, A. (2026). Tailoring calcium-alginate hydrogels: a systematic study of molecular weight and concentration effects, *International Journal of Biological Macromolecules*, 357.

Source note: Workflow adapted from the BUCHI guide, pp. 10-11; 2% alginate, 100 mM CaCl₂, and 30 min hardening are aligned with its Ca-alginate application example.

Skeletal density analysis

FIBER 101 – DEMONSTRATION PROTOCOL

Topic: Skeletal density analysis

Instrument / technique: Micromeritics AccuPyc III Gas Pycnometer

Sample: Fibrous waste material / fiber-based samples

Estimated demonstration time: 60–90 min

1. INTRODUCTION

What will we explore?

In this demonstration, we will explore the determination of the skeletal density of fibrous waste materials using gas pycnometry with the Micromeritics AccuPyc III. The technique determines the volume occupied by the solid material by measuring the displacement of gas, allowing the skeletal density of the sample to be calculated.

We will compare different sample cup volumes (10 cc, 35 cc and 100 cc) and discuss how sample quantity, morphology, packing and measurement conditions can influence the obtained result.

Learning objective

After this demonstration, you should be able to:

- understand the basic principle of gas pycnometry and skeletal density measurement;
- understand the difference between skeletal density and bulk density;
- identify the main parameters influencing the measurement of fibrous samples;
- select an appropriate sample cup volume for a given fiber material;
- interpret the measured skeletal density and assess measurement repeatability;
- recognize how skeletal density analysis can be applied to fiber-containing and waste materials.

2. THE PRINCIPLE

Gas pycnometry determines the volume of the solid portion of a sample by measuring the displacement of a gas, typically helium. The gas penetrates accessible open spaces within the sample structure, while the volume occupied by the solid material is determined from changes in gas pressure under controlled conditions. The skeletal density is then calculated from the mass of the sample divided by its measured skeletal volume.

For fibrous materials, sample morphology, fiber length, porosity, packing and the amount of sample introduced into the cup can affect the measurement. Therefore, selecting a suitable sample cup and applying consistent sample preparation are important for obtaining reliable and reproducible results.

Key concept:

Skeletal density describes the density of the solid material based on its skeletal volume, excluding the volume of accessible void spaces between particles or fibers.

Optional scheme / figure:

Sample preparation → AccuPyc III → Gas displacement / volume measurement → Skeletal volume → Skeletal density

3. MATERIALS

Sample: Fibrous waste material (brewery spent grains) and charcoal

Sample form: Fibers and powder

Amount: Selected according to the sample cup and material characteristics

Sample preparation: Homogenization and, if required, drying under defined conditions; ensure that the sample is representative and free from excessive external moisture.

Equipment:

- Micromeritics AccuPyc III Gas Pycnometer
- 10 cc sample cup
- 35 cc sample cup
- 100 cc sample cup
- Balance for sample weighing

Materials / chemicals:

- Measurement gas according to the instrument's operating procedure, typically helium
- Fibrous waste samples (brewery spent grains)
- Charcoal powder

Safety:

Follow the instrument operating instructions and laboratory safety procedures. Pay particular attention to pressurized gas and gas connections, and ensure that the sample and sample cup are handled correctly.

4. METHOD

STEP-BY-STEP PROCEDURE

1. Prepare the sample

- Obtain a representative fibrous waste sample or designated powder.
- Determine the sample mass.
- Ensure that the sample condition is suitable and consistent between measurements.

2. Select the sample cup

- Choose the appropriate cup according to the sample volume and morphology.
- Compare the use of 10 cc, 35 cc and 100 cc cups where appropriate.

3. Prepare the AccuPyc III

- Check the instrument status and measurement conditions.

- Install the selected sample cup according to the instrument procedure.
- Perform the required volume calibration / verification.

4. Run the measurement

- Introduce the sample into the cup.
- Enter the sample mass and relevant measurement parameters.
- Run the measurement according to the established method.
- Repeat the measurement where required to assess repeatability.

5. Collect and analyse the result

- Record the measured skeletal volume and calculated skeletal density.
- Compare results obtained using different sample cup volumes and/or sample preparation conditions.
- Evaluate repeatability and identify possible sources of variation.

What to observe:

- Effect of sample amount and cup size on measurement quality.
- Influence of fiber morphology and packing.
- Repeatability of skeletal density measurements.
- Differences between skeletal density and the apparent/bulk density of the fibrous material.
- Whether changing the sample cup or preparation affects the measured value.

6. RESULTS

Expected result:

The measurement provides the skeletal volume and corresponding skeletal density of the fibrous waste sample. Repeated measurements under consistent conditions should provide comparable values, while differences in sample preparation, packing, sample quantity or cup selection may influence repeatability.

How do we interpret it?

The measured skeletal density represents the density of the solid fraction of the fiber material based on its skeletal volume. A higher or lower value can reflect differences in material composition and structure, but the result must be interpreted together with the sample's morphology and preparation history. Comparison of measurements using different cup sizes can help identify suitable measurement conditions for fibrous waste samples.

Typical result / value:

To be determined experimentally for the selected fiber waste material.

8. TAKE-HOME MESSAGE

Three things to remember

1. Gas pycnometry determines skeletal volume from gas displacement and uses it to calculate skeletal density.
2. Sample preparation, sample quantity, fiber morphology and appropriate sample cup selection are important for reliable and repeatable measurements.
3. Skeletal density analysis can help characterize fiber-based waste materials and support comparison of different fiber materials, processing conditions and material compositions.

In one sentence:

Skeletal density analysis helps us characterize the solid structure of fibrous waste materials and obtain a reliable material property for their comparison and further processing.

WANT TO KNOW MORE?

Instrument / technique: Micromeritics AccuPyc III Gas Pycnometer

Further reading:

Micromeritics AccuPyc III Gas Pycnometer – instrument documentation and application information: <https://micromeritics.com/support/accupyc-iii/>

Prepared by:

AstrineXLab d.o.o.

Ljubljana, 2.9.2026

Spectrometry techniques

FIBER 101 – DEMONSTRATION PROTOCOL

Spectroscopic Techniques: From Brewery Spent Grains to Biochar

Topic: Vibrational spectrometry techniques

Instrument / technique: FTIR and Raman spectrometry

Sample: initial brewery spent grains (BSG), biochar from BSG

Estimated demonstration time: 30 min

1. INTRODUCTION

What will we explore?

Functional groups and chemical bonds in fibre-containing materials (biomass vs biochar).

Briefly explain:

- What does the technique do?
FTIR spectrometry measures infrared absorption associated with molecular vibrations to identify chemical bonds and functional groups. Raman spectrometry measures inelastic scattering of laser light to probe molecular vibrations, crystal phases, and carbon structure.
- Why is it useful for fibre research or processing?
FTIR and Raman allow us to monitor chemical and structural changes in fibres and biomass during processing, such as extraction, chemical modification, drying, pyrolysis, or carbonization.
- What information do we obtain?
The main output is a vibrational spectrum containing characteristic bands, which provide information on functional groups, phases, structure, and transformations.

Learning objective

After this demonstration, you should be able to:

- understand the basic principle of spectroscopic techniques;
- identify the main parameters influencing the measurement;
- interpret the main results;
- recognize how the technique can be applied to fibre-containing materials.

2. THE PRINCIPLE

FTIR and Raman spectroscopy are complementary vibrational spectroscopic techniques used to investigate the chemical structure and composition of materials. 1) FTIR measures the absorption of infrared radiation by a sample. Molecules absorb specific IR frequencies corresponding to their vibrations, creating a unique IR spectrum. 2) Raman spectroscopy measures the inelastic scattering of monochromatic (laser) light. When light interacts with molecular vibrations, it shifts in energy, and this “Raman shift” reveals a molecular fingerprint of the substance. Some differences are presented in Table:

Factor	FTIR spectroscopy	Raman spectroscopy
Functional groups	Polar bonds (O-H, N-N, C=O)	Non-polar bonds (C=C, aromatic rings)
Water interference	Strong absorption	Negligible
Fluorescence issue	No fluorescence interference	Can interfere
Sample preparation	Often grinding, pressing	Minimal or none
Packaging	Cannot penetrate packaging	Can analyse through glass (vials), plastic

Key concept:

Raman – prepare the instrument /switch on laser power, put the probe directly onto the sample, collect spectra, analyse the result.

ED – XRF – put the sample in XRF cup, setup the collection parameters and collect spectra

What to observe: characteristic vibrational bands (position, intensity, width).

6. RESULTS

Expected result:

FTIR: the BSG spectrum shows bands associated with O–H ($3200\text{--}3400\text{ cm}^{-1}$), C–H ($2850\text{--}2930\text{ cm}^{-1}$), C=O ($1630\text{--}1740\text{ cm}^{-1}$), C–O ($1000\text{--}1260\text{ cm}^{-1}$) and C–O–C ($1050\text{--}1150\text{ cm}^{-1}$) groups, originating mainly from cellulose, hemicellulose, lignin, proteins and other organic components. After pyrolysis, these bands decrease or disappear, indicating the decomposition of oxygen-containing and aliphatic structures.

FT-NIR: spectrum is composed of overtones and combination bands rather than fundamental vibrations. Bands associated with O–H ($5150\text{--}5250\text{ cm}^{-1}$), C–H ($4,200\text{--}4,450\text{ cm}^{-1}$), O–H / C–O ($4700\text{--}4800\text{ cm}^{-1}$), N–H ($4500\text{--}4,850\text{ cm}^{-1}$)

Raman: the biochar spectrum shows the characteristic D band and G band associated with disordered and graphitic/aromatic carbon structures. Changes in the relative intensity and width of these bands provide information about the carbon structure and degree of ordering (I_D/I_G ratio).

XRF: elemental composition, free of toxic elements

How do we interpret it?

Changes in FTIR bands indicate the loss or transformation of specific functional groups during pyrolysis, while Raman bands provide information about the formation and structural evolution of carbonaceous material. The two techniques therefore provide complementary information about the chemical and structural transformation from biomass to biochar.

Typical result / value: [if applicable]

FTIR: BSG shows characteristic bands in the rang $900\text{--}1800\text{ cm}^{-1}$ that are typical for lignin and cellulose/hemicellulose molecules.

Raman: biochar exhibits the main D band ($\sim 1310\text{ cm}^{-1}$) and G band ($\sim 1593\text{ cm}^{-1}$). I_D/I_G ratio = 1.4 indicates the formation of a relatively disordered carbonaceous structure in the biochar.

8. TAKE-HOME MESSAGE

Three things to remember

1. FTIR and Raman are complementary vibrational spectroscopic techniques that provide information about molecular and structural features of materials.

FT-NIR measures larger amount of sample, useful for initial sample assessment. Portable spectrometers used in field. Together with calibrations can be used for determining amount of key organic matter (proteins, fats, fiber and starch), moisture.

ED-XRF is very quick, non-destructive tool for measuring the elemental composition of sample

2. The quality and interpretation of the spectrum depend strongly on measurement conditions (sample properties, laser power, measurement time, sample preparation, etc.).

3. FTIR and Raman can be used to identify chemical components and monitor structural and chemical changes in fibres and biomass during processing.

In one sentence:

Spectroscopic techniques help us to identify, characterize, and monitor chemical and structural changes in fibre-containing materials.

WANT TO KNOW MORE?

Instrument / technique: <https://www.thermofisher.com/order/catalog/product/MARQMETRIXAIO>

<https://www.thermofisher.com/si/en/home/industrial/spectroscopy-elemental-isotope-analysis/molecular-spectroscopy/fourier-transform-infrared-spectroscopy/instruments/nicolet-is50-ftir-spectrometer.html>

<https://elvatech.com/products/elvax-plus/>

Further reading:

<https://www.irdg.org/internet-journal-of-vibrational-spectroscopy-archive>

<https://www.linkedin.com/pulse/raman-spectroscopy-vs-ftir-whats-best-you-technos-instruments-gs3sc>

<https://www.thermofisher.com/si/en/home/industrial/spectroscopy-elemental-isotope-analysis/molecular-spectroscopy/fourier-transform-infrared-spectroscopy/resources/ftir-spectroscopy-academy.html>