









Rheology and physical measurements for the characterization of foods and materials

Able to link the rheological and intrinsic mechanical properties of materials to their technological performance.

Denis
Cassan

U.M.R. 1208 Ingénierie des Agropolymères et des Technologies Emergentes (I.A.T.E.) 2, place Viala 34060 MONTPELLIER Cedex 01 Tel: 04 99 61 31 46

Denis.Cassan@supagro.inra.fr http://www.montpellier.inra.fr/umr-iate/ 2013

<u>Summary</u>

Rheological and thermal analysis

ARES - Advanced Rheometric Expansion System - Rheometric Scientific	Page 2
AR2000ex – TA instruments	Page 3
Physica MCR 300 - Anton Paar	Page 4
Tritec200B - Triton Technology Ltd	Page 5
DMTA 3E - Rheometric Scientific - Piscataway, USA	Page 6
FT4 - Freeman Technology	Page 7
Capillary viscometer	Page 8
DSC Q200 - Differential Scanning Calorimeter – TA Instruments	Page 9
Toutand and built for Josefanials	
Textural analysis: food and materials	
Textural analysis: food and materials Ta.XT Plus – Stable Micro Systems	Page 10
	Page 10
Ta.XT Plus – Stable Micro Systems	_
Ta.XT Plus – Stable Micro Systems Powder flow Analyser – Stable Micro Systems	Page 11
Ta.XT Plus – Stable Micro Systems Powder flow Analyser – Stable Micro Systems Ultrapycnometer 1000 – QUANTACHROME	Page 11
Ta.XT Plus – Stable Micro Systems Powder flow Analyser – Stable Micro Systems Ultrapycnometer 1000 – QUANTACHROME RVDV-II+PRO - BROOKFIELD	Page 11 Page 12 Page 13 Page 14
Ta.XT Plus – Stable Micro Systems Powder flow Analyser – Stable Micro Systems Ultrapycnometer 1000 – QUANTACHROME RVDV-II+PRO - BROOKFIELD Plasticorder PL 2100 – Brabender	Page 11 Page 12 Page 13 Page 14
Ta.XT Plus – Stable Micro Systems Powder flow Analyser – Stable Micro Systems Ultrapycnometer 1000 – QUANTACHROME RVDV-II+PRO - BROOKFIELD Plasticorder PL 2100 – Brabender Plastograph – Brabender	Page 11 Page 12 Page 13 Page 14 Page 15

ARES

Advanced Rheometric Expansion System, Rheometric Scientific

A rheometer is a mechanical spectrometer that is capable of subjecting a sample to either a dynamic or a steady shear strain and do measure the resultant torque exerted by the sample in response to the shear strain.



- (1) Screen control
- (2) Manual Control
- (3) Geometries
- (4) Peltier Plate.
- (5) Control knob to raise or lower the tool. The middle button double speed

ARES is shear strain controlled rheometer: shear strain is applied by the motor; torque is measured by the transducer.

The temperature is controlled by Peltier.



4 geometries available:

- o Cone-Plate
 - Diameter 50 mm
 - Diameter 25 mm
- o Plate-Pate
 - Diameter 50 mm
 - Diameter 25 mm

Specifications:

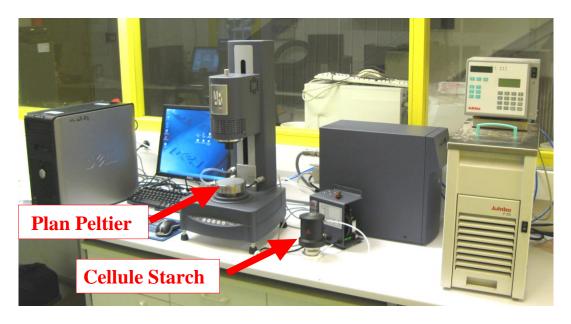
Temperature: -30°C à +150°C (30°C/minute de 0°C à 100°C)

Torque : de 2 à 2000 g.cm

Angular displacement: de 0,05 à 500 milli radians Speed: 1 E-5 à 500 rad/sec avec une résolution de 0,098%

AR2000ex – TA Instruments

AR200ex is stress controlled rheometer. Shear stress is applied by the motor located in the upper part. An optical sensor accurately measures the axis rotation induced by the deformation of the sample under effect of the stress.



This rheometer allows measurements in flow, stress relaxation, oscillation, and normal force.

Spécifications:

Torque Range: 0.1 μ Nm to 200 mNm Frequency Range: 7.5 E-7 to 628 rad s-1 Angular Velocity Range: Controlled Stress: 0 to 300 Rad s-1

Controlled Strain: 10-8 to 300 Rad s-1

Angular Displacement: 40 nRad

Resolution Minimum Strain: 0.00006 Normal Force Range: 0.005 to 50 N

Geometries available:









Temp range -20°C to 200°C
Heatin rat max : 20°C/min
Various Plates
Various Cones

Peltier Plate

Starch Pasting Cell

Temp range 5°C to 120°C Heatin rat max : 30°C/min

Concentric Cylinder

Temp range -10°C to 150°C Heatin rat max : 15°C/min

+ System VANE

Pressure Cell

Temp range -10°C to 150°C Pressure 140 bar

Physica MCR 300 - Anton Paar



Any type or combinations of rheological tests, both in rotational and oscillatory mode, are possible with the MCR 300.

Accessories:

- Peltier plate
- Peltier concentric cylinder
- Pressure cell, paper coating cell

Specifications:

Plage de couple : $0.02~\mu Nm$ à 150~mNm

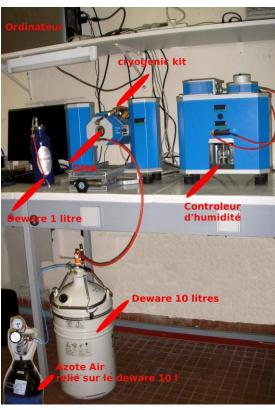
Plage de déplacement angulaire : 0.1 µrad à infini

Plage de fréquence : 10⁻⁴ à 100 Hz Vitesse pilotable : 10⁻⁵ à 300 rad/s Vitesse mesurable : 10⁻⁸ à 300 rad/s

Force normale mesurée et appliquée : 0.01N à 5N

Dynamic Mechanical Analyser TRITEC200B Triton Technology Ltd





This DMA enable to study the mechanical properties of materials under a variety of conditions from temperature and humidity controlled.

This instrument allows for different experiences depending on the selected geometry: single and dual cantilever bending, tension, compression, shear, and 3-points bending

Specifications:

Environmental Conditioning: Immersion or Humidity control

Temperature Range: -190°C to 400°C

Heating and Cooling Rates: Heating rate 0.01 to 20°C/min, Cooling rate 0.01 to 40°C/min

Frequency: Range 0 to 300Hz, Max. Number Up to 100 per experiment, Precision 0.001Hz

Dynamic Displacement: $0 \text{ to } \pm 1000 \mu\text{m}$

Stiffness: 2 x 102 to 1 x 108 N/m

Modulus: Resolution 0.0001 Pa, Range 1 x 102 to 7.5 x 1014 Pa (Theoretical)

Tan Delta: Resolution 0.00001

Force: Range \pm 10N, Minimum 0.002N, Resolution 0.0001N, Strain: Resolution 1nm, Max. Number 100 per experiment

Sample size: Maximum 52.5mm x 12.8mm x 8.0mm

$\begin{array}{c} \textbf{Dynamic Mechanical Thermal Analysis} \\ \textbf{DMTA 3E} \end{array}$

Rheometric Scientific

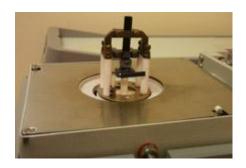
Piscataway, USA

According to a sinusoidal movement, viscoelastic samples are sought in tension or in flexion or shear in a temperature controlled (oven), measuring the amplitude and phase shift of the resultant stress.

The stress is in phase with the strain for a perfectly elastic material.

Thermomechanical spectra are plotted (G' et G'' ou tan δ).

Samples must be analyzed in the linear domain (proportional deformation and strength).



Specifications:

Frequency range	[rad/sec]	0,01 100
Amplitude range	[mm]	-0,05 0.05
Tensile resolution		-0,05*10-6 0,05*10-6
Temperature range	[°C]	-150 600
Temperature modification	n [K/min]	0,1 50
Phase angle resolution	[Grad]	-0,1 0,1
Maximum force (at 1kg)	[N]	9,81
Measurement sensitivity	[g]	1



Powder analysis FT4 - Freeman Technology



The FT4 Powder Rheometer is a universal powder tester - three instruments in one, combining the Freeman patented blade methodology for measuring flow energy with a range of shear cells, wall friction modules and other accessories for measuring bulk properties.

The methodologies allow measurement of:

- Flow energy in relation to many variables and all packing states
- Shear properties of consolidated and unconsolidated powders
- Bulk properties precision bulk density, compressibility and permeability
- Process variables such as moisture, attrition and segregation



The System accessories available include:

- Three sizes of testing vessel: 25, 50 and 62mm diameter for different samples sizes.
- Aeration Control module to aerate/fluidise powder samples
- Compaction accessories for consolidating powder
- Shear cell modules three sizes down to the 1ml cell
- Wall friction modules to measure powder on metal interaction







Capillary viscometer



The solution is placed in a capillary tube type Ostwald (Fig1)

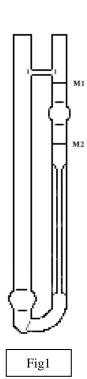
The liquid is drawn above point M1, and then the decay time of the liquid is measured between M1 and M2, with two photocells.

The measurement is made under controlled temperature (20 $^{\circ}$ C - 50 $^{\circ}$ C).

The decay time of the solution is assimilated to viscosity.

3 sizes of tubes available: fine, medium, big.

Measurement of solution viscosity between 0.5mPa et 5mPa



Differential Scanning Calorimeter DSC Q200 – TA Instruments



Differential scanning calorimetry or DSC is a thermoanalytical technique in which the difference in the amount of heat required to increase the temperature of a sample and reference is measured as a function of temperature.

A modulated temperature differential scanning calorimeter (MTDSC) can be used in the Q200, in which a small oscillation is imposed upon the otherwise linear heating rate.

Specifications:

Temperature Range Ambient to 725 °C With cooling accessories -40 to 400 °C Temperature Accuracy +/- 0.1 °C Temperature Precision +/- 0.05 °C Calorimetric Reproducibility (indium metal) +/- 0.1 % Calorimetric Precision (indium metal) +/- 0.1 % Baseline Curvature (Tzero; -50 to 300 °C) 10 μW Baseline Reproducibility with Tzero +/- 10 μW Sensitivity 0.2 μW Indium Height / Width (mW/°C)* 30



Tzero DSC Sample Encapsulation Press



The RCS40 employs a single-stage refrigeration system, which permits convenient DSC operation over the temperature range from -40 °C to 400°C.

Textural analysisTa.XT Plus – Stable Micro Systems



The texture analyser TA.XTplus (Stable Micro Systems) is able to measuring virtually any physical product characteristic such as hardness, fracturability, adhesiveness, gel strength, extensibility of foods, cosmetics, pharmaceuticals, gels, adhesives and other consumer products.



Specifications:

Force Capacity: 30kg.f (300N) Force Resolution: 0.1g Speed Range: 0.01 – 40mm/s Range Setting: 0.01 - 280mm



Available blades : (see pictures)



Powder analysis Powder flow Analyser – Stable Micro Systems



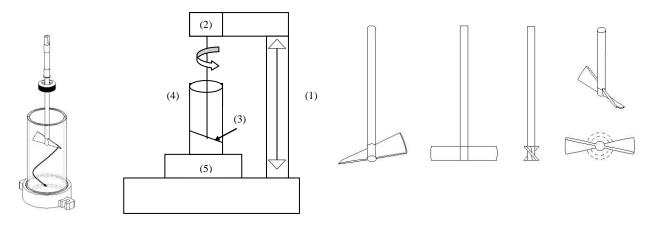
On the texture analyser TA.Xtplus de Stable Micro Systems Ltd, we can attach the Powder Flow Analyser to extend our range of testing solutions from powders to end products.

This module uses the vertical drive system (1) of the texture analyzer. It is composed of a system of rotation (2), a specific spiral blade (3), a vertical cylindrical tank (4) and a vertical force sensor (5) located under the vessel.

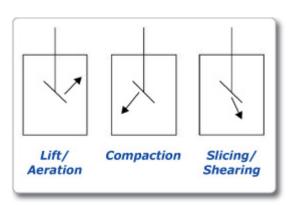
Normal force : 50 N (\pm 0.05%)

Blade tip speed: A 0 deg: 0 to 754mm/sec (limit of 300rpm rotational speed)

A 90 deg: 0 to 40mm/sec



Depending upon the programmed rotation of the blade in terms of path angle, blade direction and tip speed the powder column can be displaced in a number of different ways to suit the process and properties to be measured.



Pycnomètre à gaz Ultrapycnometer 1000 - QUANTACHROME

"Pycnometry" is derived from the Greek word puknos, which has long been identified with volume measurements. This pycnometer is specifically designed to measure the true volume of solid materials by employing Archimedes' principle of fluid displacement and gas expansion (Boyle's Law).





Standard sample cells 135 cm3 50 cm3 10 cm3

The Ultrapycnometer is an instrument for measuring the true volume and density of powders, foams and bulk solids.

A wide range of sample cell sizes are easily interchanged to accommodate many different samples. Calibration, sample conditioning, operation of valves and calculation of results are completely automatic



Calibration Set

BROOKFIELD RVDV-II+PRO Viscometer



This instrument allows the measurement of the viscosity and temperature simultaneously.

Specifications:

Viscosity : de 100 à 40 millions Centipoise. Speed: de 0,01 à 200 rpm Torque accuracy: 1% Repeatability : 0,2 %

Plasticorder PL 2100 - Brabender

Plasticorder is a torque rheometer for applicational investigations or processing tasks in laboratories and for simulation. This instrument is suited for mixers and extruders including a temperature control.

Plasticorder PL 2100:

Speed: 0 - 120 min⁻¹ Torque: max. 200 Nm



Mixer W 50:

Speed: 0 - 120 min⁻¹ Temp: max. 300 °C Torque: 0 - 200 Nm Volume: 55 cm³



Mixer W 50



Extrudeur DSE25



Plastograph – Brabender





Plastograph is a torque rheometer (0-100 Nm, speed 0-150 min⁻¹). Equipped with a planetary mixer (P600), it can be used for suspensions study.

Also, Farinograph® can be used with 50 g of flour.



Mixograph - National Mfg. Co.



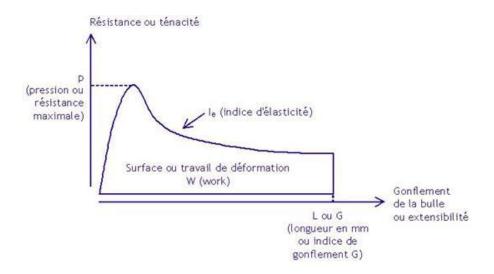
The Mixograph is a recording dough mixer that quickly analyzes flour for gluten strength, proper absorption, mixing peak time and mixing tolerance. (Available in 10 gram formats, computer recording)

Alvéograph - CHOPIN



The Chopin alveograph is a tool for flour quality measurement.

It measures the flexibility of the dough produced from the flour, by inflating a bubble in a thin sheet of the dough until it bursts. The resulting values show the strength of the flour, and thus its suitability for different uses.



Rapid Visco Analyser RVA - 4 Newport Scientific

The RVA is a rotational viscometer measures the viscosity gelatinization of the starch.

Temperature Range: 0 - 99.9°C

Temperature Accuracy: +/- 0.3°C at 25°C Heating/Cooling rate: up to 14°C/minute

(Infinitely variable)

Speed Range: Computer controlled Infinitely variable 10 - 2000 rpm Speed Accuracy: +/- 1% at 160 rpm

Viscosity Range: 50 -50,000 cP at 80 rpm

Viscosity Accuracy: +/- 3% for S2000 Oil nom. 5000 cP

