LUM

Analytical instruments for the entire life cycle of batteries, energy materials & more...

Direct, accelerated & real-time stability of battery slurries in all processing stages

Hansen Parameters (HSP/HDP)
Particle dispersibility

Determination of particle size distributions & particle concentrations

Tensile & shear testing of electrode coatings, Cu foils and PTFE substrates

Key control characteristics for energy materials

Though active materials for batteries and fuel cells expand across the whole periodic table, the need of carbon (carbon black, graphene, carbon nanotubes, …) is not debatable. When dispersing, e.g., carbon black in a distributive or disruptive process, three independent factors influence the success:

1. The inherent chemical and physical properties of the raw materials, for example, dry or wet, particle size and size distribution, shape, brittleness or state of agglomeration.
2. The interaction between the particle surface and the continuous phase.
3. The dispersion process itself. Time, energy, intensity and mechanism (mechanical principle) have an influence on the success of the distributive or disruptive process.

Therefore, to describe dispersibility all three factors must be considered (multidimensional approach).
In later process stages, the dispersion stability or separation stability of the battery slurries needs to be characterized in original concentration.

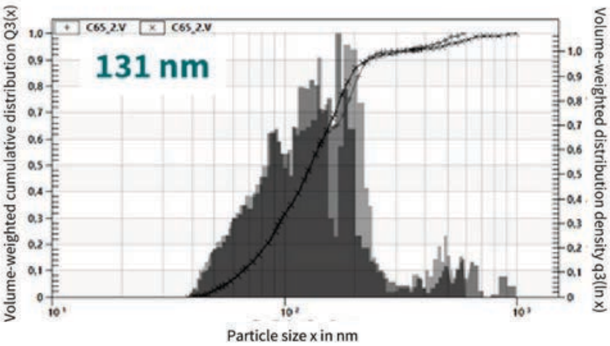
Based on the optical STEP technology and the multi-sample concept, the LUM analysers allow for the efficient determination of particle size distributions and particle surface properties according to the Hansen solubility/dispersibility parameter concept.

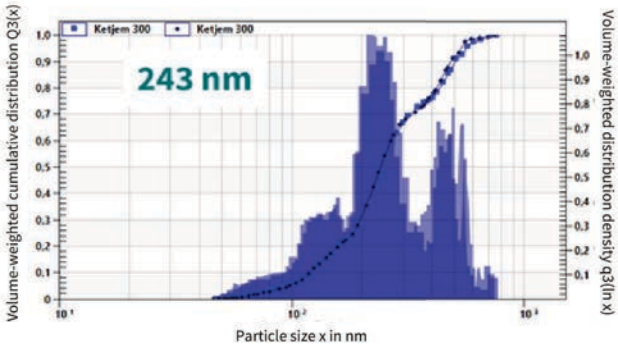
Manufactured battery pastes are characterized undiluted with regard to their separation stability using complementary X-ray STEP technology.

Multi-wavelength Dispersion Analyser

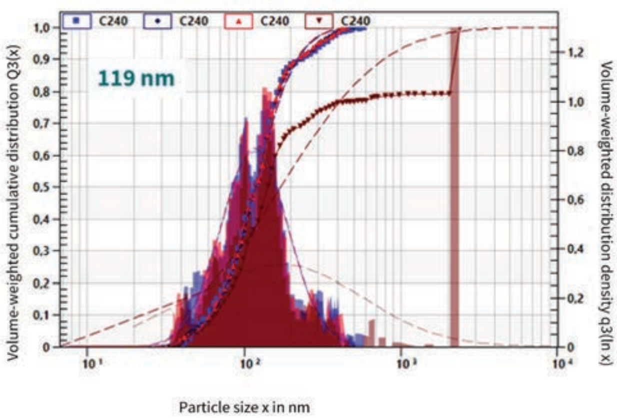
- Run direct & accelerated stability measurements in original concentration
- Particle size distribution (PSD) with high resolution
- Study up to 12 samples simultaneously
- See & understand complete samples from top to bottom
- Measure samples in a broad temperature range (4 °C to 60 °C), constant or ramping
- Measure particle size even at higher concentrations
- Analyse concentrated samples (up to 90Vol%)
- Acquire particle densities
- HSP/HDP
- Norms: ISO 13318, ISO/TR 13097, ISO/TR 18811, ISO 18747

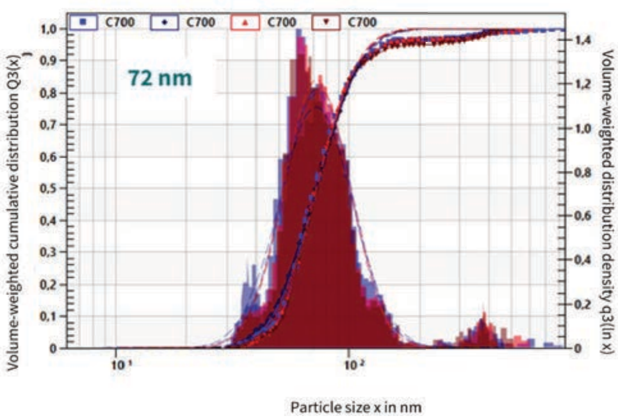
Particle Size Distributions of Carbon Black





Particle Size Distribution of platinized Carbon Black





Particle Dispersibility: Impact of preparation route (type, energy, time) on surface properties of Carbon Black (HDP)

	Dispersing by	HDP (δ_D δ_P δ_H) [MPa$^{1/2}$]			Best PL and its HSP-values (δ_D δ_P δ_H) [MPa$^{1/2}$]			
A	vortex	17.1	12.1	12.5	DMSO	18.4	16.4	10.2
B	2x 15 min ultrasonic bath	16.8	10.0	9.2	DMF	17.4	13.7	11.3
C	ultrasonic bath + 1 min sonotrode (67 J/cm^3)	17.6	7.7	11.4	Aceton	15.5	10.4	7.0
D	ultrasonic bath + 10 min sonotrode (670 J/cm^3)	17.0	12.2	0.1	ACN	15.3	18.0	6.1

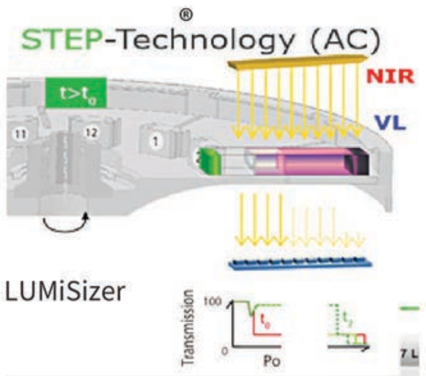
Steps for determination of Hansen Parameters (HSP/HDP) of Carbon Black

Step 1:
Selection of suitable solvents (PL) from the 3D Hansen range.

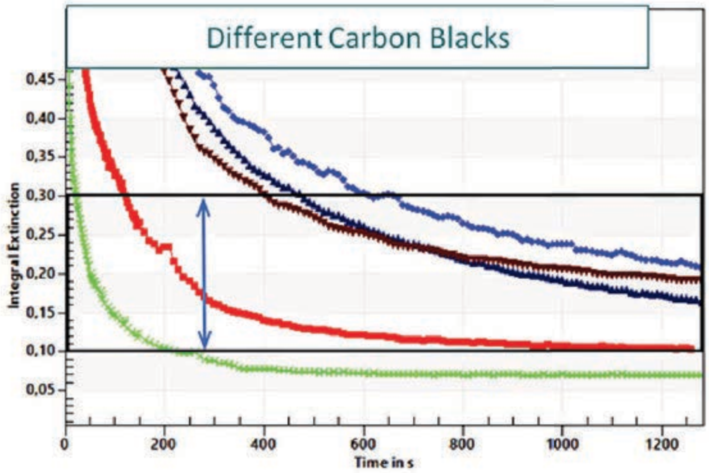
PL	Abbr.	δ_D [$MPa^{1/2}$]	δ_P [$MPa^{1/2}$]	δ_H [$MPa^{1/2}$]	V [cm^3/mol]	ρ (kg/m^3)	$\rho_{CB} - \rho_{PL}$ (kg/m^3)	η [mPa/s]	Purity %	Boiling temp.
Dimethyl sulfoxide	DMSO	18.4	16.4	10.2	71.3	1100	700	1.8	99,5	189
N-Methyl-2-pyrrolidone	NMP	18.0	12.3	7.2	96.6	1020	780	1.52	99,8	202
Dimethylformamide	DMF	17.4	13.7	11.3	77.4	945	855	0.802	99,8	153
2-Butoxy ethyl acetate	BuOAc	15.3	7.5	6.8	136.1	940	860	1.7	98	192
2-Propanol	IPA	15.8	6.1	16.4	76.9	786	1014	1.77	99,95	82
Tetrahydrofuran	THF	16.8	5.7	8.1	81.9	890	910	0.54	99,5	66
Ethyl acetate	EtAc	15.8	5.3	7.2	98.6	900	900	0.44	99,5	77
1.4-Dioxane	Diox	17.5	1.8	9.0	85.7	1030	770	1.17	99,5	101
Acetone	Ace	15.5	10.4	7.0	73.8	779	1021	0.3	99,8	56
Acetonitrile	ACN	15.3	18.0	6.1	52.9	786	1014	0.33	99,95	82
Triethylamine	Tri	15.5	0.4	1	139.7	728	1072	0.347	99,5	89
n-Hexane	Hex	14.9	0	0	131.4	659	1141	0.30	99	69
Dimethyl-carbonate	DMC	15.5	8.6	9.7	84.7	1069	731	0.664	99,8	90
Methanol	MeOH	14.7	12.3	22.3	40.6	782	1018	0.507	99,95	65

Step 2:
Dispersing Carbon Black in the selected solvents.

Step 3:
Analysis of separation behavior by analytical centrifugation (AC)

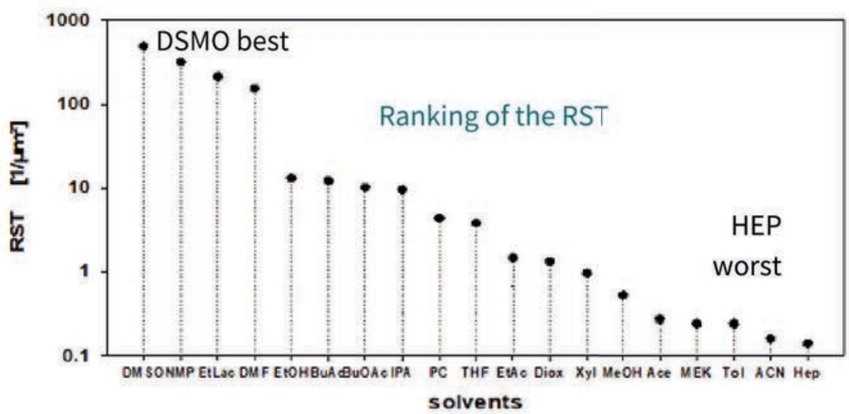


Step 4:
Determination of the sedimentation time (ST_i) from the Integral Extinction for selected region & interest



Step 5:
Determination of the relative sedimentation time

$$RST_i = \frac{ST_i\,(\rho_P - \rho_{l,i})\,RCA\,g}{\eta_{l,i}\,d_{cell}}$$



X-Ray Separation Analyser

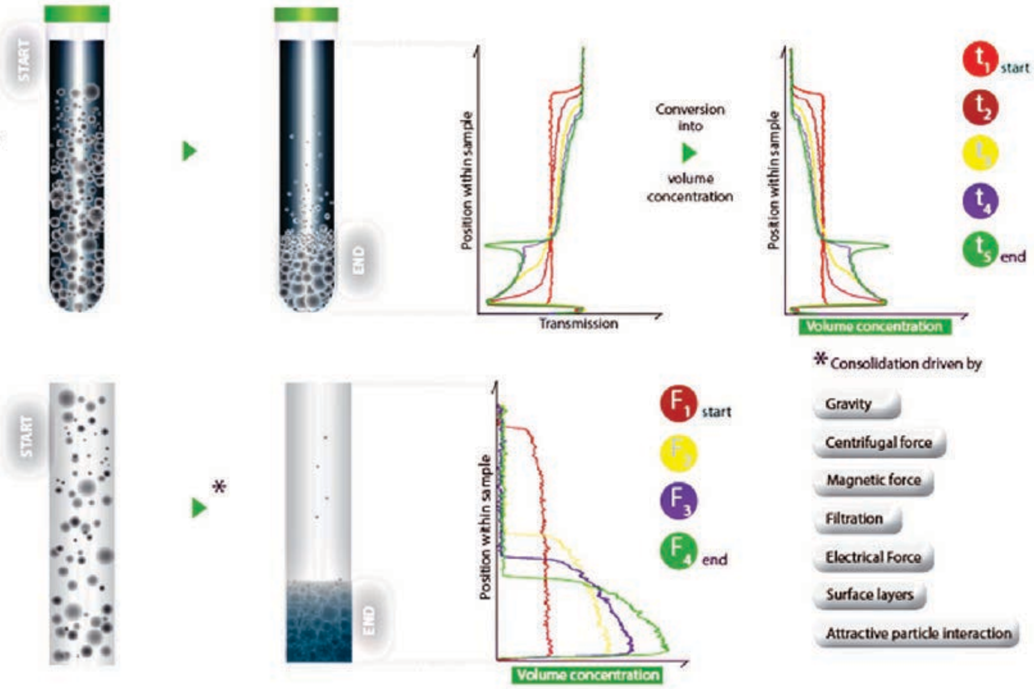
- Analyse dispersibility, stability and separation phenomena for completely transparent to completely opaque emulsions, suspension, sludges, slurries, foams and powders in real-time
- Characterization of battery pastes in different process stages by means of X-Ray
- In-situ sediment & cake structure analysis
- Solidosity, dewaterability & consolidation analysis
- High resolution phase separation
- Detect concentration gradients within phases and sediment
- Norms: ISO 18747, ISO/TR 13097, ISO/TR 18811

STEP-Technology - LUMiReader X-Ray

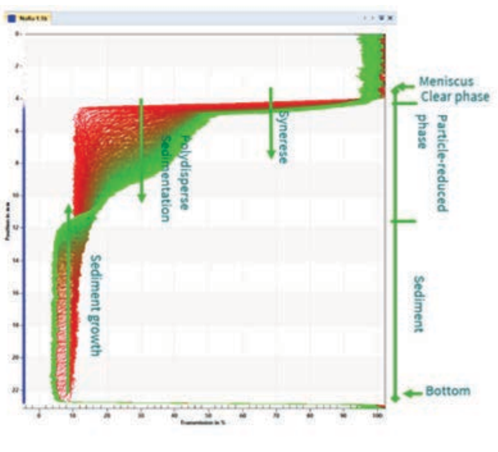
1. Separation in real-time – Battery pastes

2. Before & after accelerated separation with a LUMiSizer

Fingerprints of the consolidation

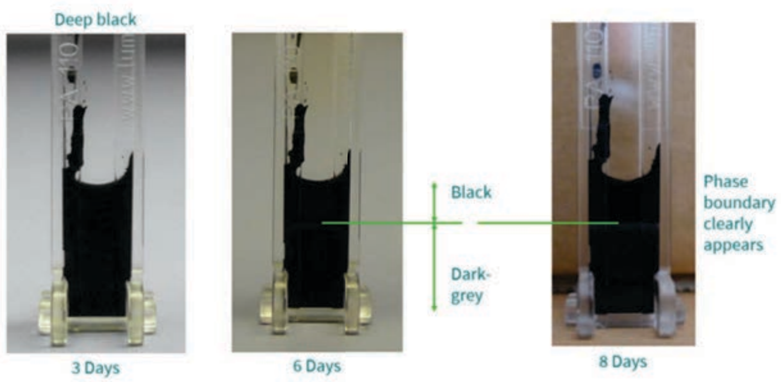


Real-time separation of the battery pastes with X-Ray



Sample 1.1b: 400 profiles every 120s = 0,5 days

Visual appearance after storage at 1 g



Application of the LUMiFrac® for testing the adhesive strength of cathode & anode coatings in lithium-ion batteries

In a lithium-ion battery, the smallest unit of each cell consists of two electrodes (anode and cathode), and the separator. The ion-conducting electrolyte is located in between. Typically, the main component of the anode is graphite, which is excellent for storing lithium ions during charging.

However, graphite's limited electrical conductivity can cause inefficiencies in charge and discharge cycles. To boost the anode's performance, the coating system is applied to a thin copper foil.
The copper foil enhances electrical conductivity which facilitates the electron flow between the anode and the rest of the battery, including the cathode. As a result, the battery's internal resistance decreases, leading to more efficient charging and discharging processes.

This translates to higher power output and better overall performance. The coating system prevents corrosion of the copper foil, ensures stability, and maintains electrolyte compatibility.

The active material of a cathode consists largely of a lithium transition metal oxide compound, like lithium cobalt oxide or lithium iron phosphate. While these materials have good energy storage capabilities, they can be chemically reactive and degrade over time.

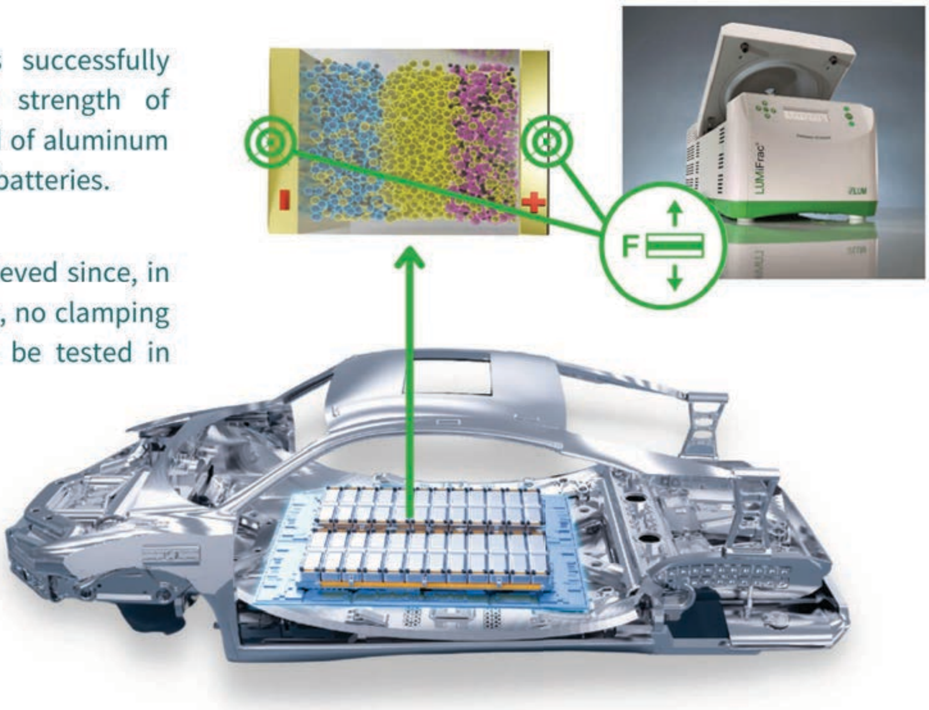
To address this concern and improve stability and lifespan, the cathode coating system is applied to a thin aluminum foil. This foil and the corresponding coating system reduce or prevent undesirable side reactions and chemical degradation, ensuring the battery's long-term stability and cycle life.

The coating process can be achieved using various techniques, such as solvent casting, extrusion coating, or lamination, depending on the specific requirements and manufacturing processes of the lithium-ion battery.

Overall, the coating system on the aluminum foil plays a vital role in ensuring the stability and safety of the cathode in a lithium-ion battery and preventing dendrite formation.
The adhesive strength of the coatings on the metal foils is one vital indicator for the overall performance of the lithium-ion battery.

The adhesion analyser **LUMiFrac®** is successfully utilized to determine the adhesive strength of coatings on copper foils for anodes and of aluminum foil coatings for cathodes in lithium-ion batteries.

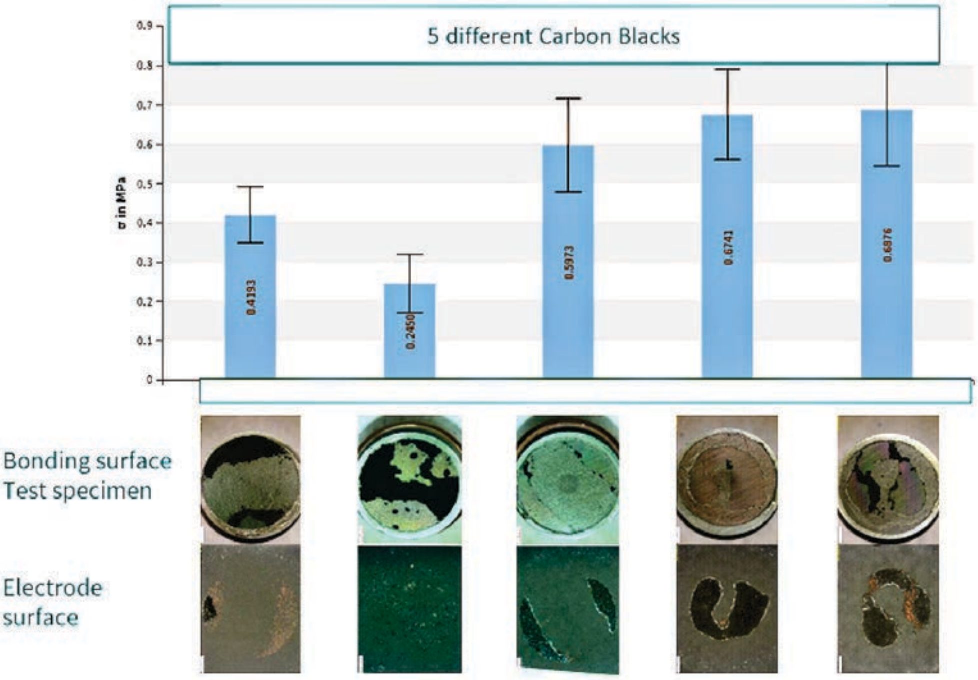
A time saving of up to 80% can be achieved since, in contrast to other comparative methods, no clamping is required and up to 8 samples can be tested in parallel.



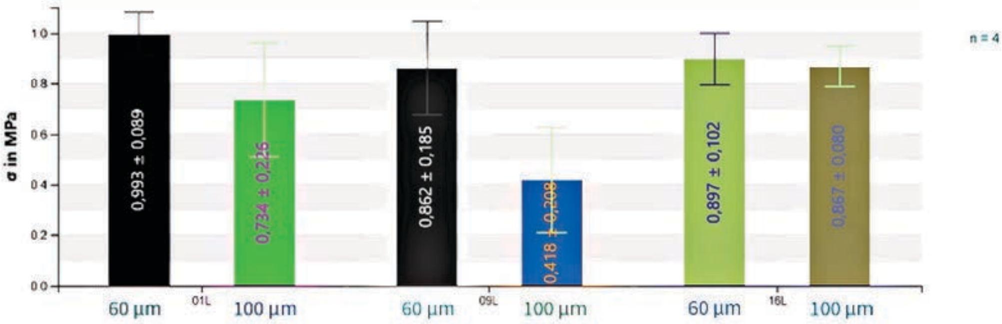
Adhesion Analyser

- Adhesion strength of coatings on Cu-foils for battery electrodes, coated PTFE and PFSA substrates for fuel cells
- Comparison of sample groups and production batches
- Tensile, shear & bonding strength
- 8 different samples @ identical conditions
- No sample clamping, any substrate
- Increasing & alternating loads (0.1 N-6.5 kN)
- Cost-saving multi-use of test stamps
- Temperature controlled (-11 °C to +40 °C)
- Norms: ISO 4624, DIN EN 15870, JIS K 5600-5-7, DIN EN 13144, ASTM D4541, DIN EN 14869-2, ISO 9211-4

Bonding strength of tested carbon black & electrode foil coating samples



Strengths of the manufactured electrodes



	60 µm	100 µm
01L	0,993 ± 0,089	0,734 ± 0,226
09L	0,862 ± 0,185	0,418 ± 0,208
16L	0,897 ± 0,102	0,867 ± 0,080

LUMiSpoc® Single Particle Optical Counter

- Particle counting & size distribution
- Number concentration determination
- Direct number-based particle size distribution of nano- & microparticles
- Classification of nanomaterials

Norms: ISO 21501-2

More info on LUMiSpoc.com

Further Applications (1/3)

Lithium manganese phosphate - Particle Size Distribution according to ISO 13318-1 and ISO 13318-2 – Request the application note via support@lum-gmbh.de

Negative active material for rechargeable lithium battery, method of preparing the same, and negative electrode and rechargeable lithium battery including the same - https://bit.ly/3LO2Rcf

A new approach to characterization of particle surface properties by means of analytical centrifugation - https://bit.ly/3LJWRBr

Comparison of carbon onions and carbon blacks as conductive additives for carbon supercapacitors in organic electrolytes - https://bit.ly/3KKqQsb

Organosol composition of fluorine-containing polymer - https://bit.ly/3H7t9EO

Next generation electrochemical energy storage with nanocarbons and carbon nanohybrid materials - https://bit.ly/3O0bJOG

Indirect tuning of the cathodic PEMFC electrode microstructure and its functionality for automotive application - https://bit.ly/3Me5Ags

Dispersant for separator of non-aqueous electrolyte battery comprising 2-cyanoethyl group-containing polymer and separator and battery using the same - https://bit.ly/42oKJN5

Structure and Properties of Supercapacitor and Lithium-Ion Battery Electrodes: The Role of Material, Electrolyte, Binder and Additives - https://bit.ly/3LV2LRY

Redox electrolytes for non-flow electrochemical energy storage - https://bit.ly/3M17n8Y

LUMiReader® PSA Separation Analyser

- Real-time stability directly
- Speed up separation analysis time (up to 10-fold)
- Volume and number-based PSD (ISO 13317)
- Volume-based PSD without refractive indices
- Multi-wavelength approach
- Separation velocity distribution
- Temperature stabilization from 4 °C - 80 °C, constant or ramp
- Any dispersing media: water, oils, organic solvents

Norms: ISO/TR 18811, ISO 13317, ASTM D7827, ISO 18747, ISO/TR 13097

More info on LUMiReader.com

Further Applications (2/3)

Method for manufacturing separator, separator manufactured thereby, and electrochemical device comprising same - https://bit.ly/3VP9jEm

Analysis of Wireless Body-Centric Medical Applications for Remote Healthcare - https://bit.ly/436yF46

Separation membrane for secondary battery and electrochemical element to which it is applied - https://bit.ly/42pgTbn

Tailored SiN -based Anode Processing for Li-Ion Batteries - https://bit.ly/3NXJb8K

Slurry composition for coating secondary battery separator and secondary battery separator using same - https://bit.ly/3B9GMQF

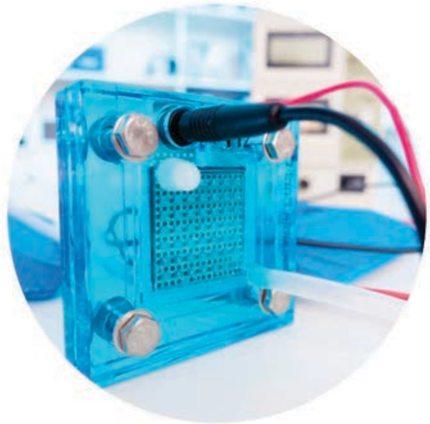
A Hybrid Electrochemical Energy Storage Device Using Sustainable Electrode Materials - https://bit.ly/42ofHF4

On the state and stability of fuel cell catalyst inks - https://bit.ly/3VPie8O

CO2/CH4 and He/N2 Separation Properties and Water Permeability Valuation of Mixed Matrix MWCNTs-Based Cellulose Acetate Flat Sheet Membranes: A Study of the Optimization of the Filler Material Dispersion Method - https://bit.ly/3piP5Xu

On the drying kinetics of non-spherical particle-filled polymer films: A numerical study - https://bit.ly/42F5ZxW

Effect of the Anionic Counterpart: Molybdate vs. Tungstate in Energy Storage for Pseudo-Capacitor Applications - https://bit.ly/44F5cyO



Further Applications (3/3)

Systematic evaluation of materials and recipe for scalable processing of sulfide-based solid-state batteries - https://bit.ly/3HY2ACi

Upgrading the Properties of Ceramic-Coated Separators for Lithium Secondary Batteries by Changing the Mixing Order of the Water-Based Ceramic Slurry Components - https://bit.ly/3MbVFYO

Synergistic Effect of Dual-Ceramics for Improving the Dispersion Stability and Coating Quality of Aqueous Ceramic Coating Slurries for Polyethylene Separators in Li Secondary Batteries - https://bit.ly/40okrdo

Dopant Interaction in Binary Metal Oxide System: Towards the Development of an Improved Supercapacitor Material - https://bit.ly/42EW2R8

Catalyst Preparation and Particle Size Characterization Scheme for PEMFC Fuel Cells - https://bit.ly/3HThMk7

Investigating the Effect of Solvent Composition on Ink Structure and Crack Formation in Polymer Electrolyte Membrane Fuel Cell Catalyst Layers - https://bit.ly/3VSswFj

Auf dem Weg zur Lagerstabilitätsvorhersage nach I50/TR 13097- ein Beispiel für die schnelle Prüfung der einfachen Anwendbarkeit physikalischer Beschleunigung - https://bit.ly/3pvnPVK

Nanostrukturierte polyionische Flüssigkeiten auf Basis hyperverzweigter Polyoxetane als Transporter, Dispergiermittel und Hybridmaterialien - https://bit.ly/3lMf8ow

Elektrodenmaterialien für organische Energiespeicher auf Basis elektrochemisch aktiver Polymere und Graphen - https://bit.ly/41Bxw3e

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Demo & samples

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T +49 30 6780 60 30

M support@lum-gmbh.de

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LUM knowledge

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bit.ly/3J1mUUS