

Introducing The *MagnoMeter*[™] XRS

A new Instrument for
Particle Characterization and Surface
Analysis of Dispersions

What is a *MagnoMeter*?



- ⦿ A device that uses NMR relaxation for non-invasive routine analysis of complex solid-liquid and liquid-liquid formulations
 - ⦿ Samples in water, solvents and melts
 - ⦿ No dilution required, minimal sample preparation

- ⦿ Ideal for applications where speed of measurement and reliability are drivers of performance
 - ⦿ Research & development
 - ⦿ Quality control/quality assurance
 - ⦿ Process control labs

What is NMR relaxation?



- ⌘ NMR relaxation time is a fundamental, intrinsic property of all solids and liquids
 - ⌘ Analogous to intensity of scattered light (particle sizing) and electrophoretic mobility (zeta potential)
- ⌘ Relaxation time of suspensions is intermediate between that for solid and liquid
 - ⌘ Value depends on specific particle-liquid combination
 - ⌘ Determined directly using an NMR spectrometer

Why use NMR relaxation?



“ In every industrial application, a knowledge and understanding of the molecular structure and dynamics at the particle-liquid interface is critical to improving, or optimizing, suspension and emulsion product performance.”

NMR Instruments



High resolution NMR

well-known technique for studying molecular structure and identification of compounds



- ⦿ high frequency needed
- ⦿ expensive, complex, sophisticated operation
 - ⦿ intensive training
- ⦿ university and analytical laboratories

Low field NMR

new technique for suspension and emulsion analysis



- ⦿ low frequency optimal
- ⦿ inexpensive, simple benchtop device
 - ⦿ easy operation
- ⦿ industrial R&D, QC/QA and Process laboratories

What can you measure?



- ⌘ Dispersed (wetted) Surface Area (suspensions)
- ⌘ Dispersed particle volume fraction
- ⌘ Molecular weight (polymers in solution)*
- ⌘ Polymer and solvent viscosity
- ⌘ Particle Size (<30nm)
- ⌘ Relaxation Number (liquids/suspensions/emulsions)
 - ⌘ Kinetic processes
 - ⌘ Adsorption/desorption
 - ⌘ Competitive adsorption
 - ⌘ Colloidal stability
 - ⌘ Presence of para- and ferro-magnetic impurities
 - ⌘ Hydroxyl (OH) number of metal oxides
 - ⌘ Oxygen and water content of solvents

* By calibration

“ The MagnoMeter can be used in an almost unlimited range of applications, and can measure samples at virtually any industrially relevant solids concentration.”

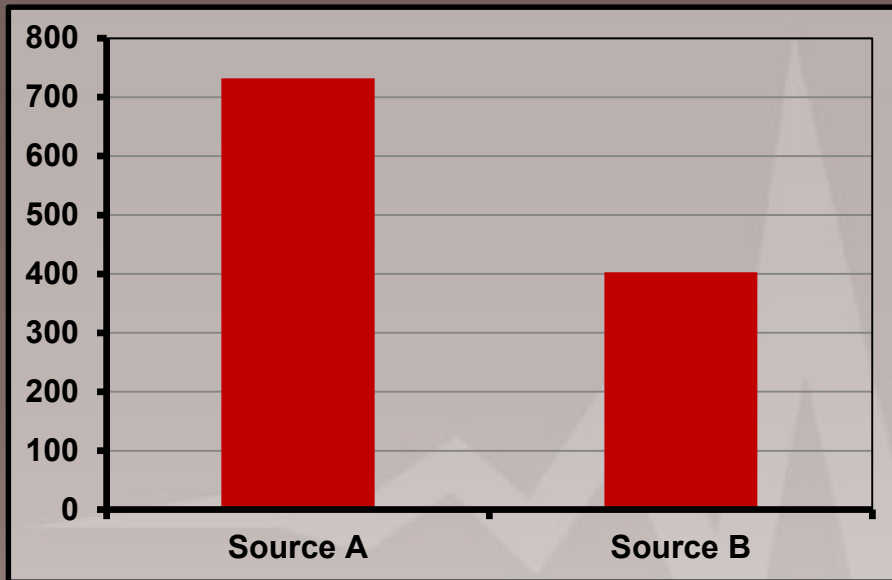
- ⦿ Graphene/Graphene Oxide
- ⦿ Cellulose nanocrystals
- ⦿ Ceramics, refractories
- ⦿ Minerals, metal oxides
- ⦿ Paints and inks
- ⦿ Dyes
- ⦿ Pharmaceuticals

- ⦿ Nanomedicine
- ⦿ Cosmetics
- ⦿ Food emulsions
- ⦿ Agrochemicals
- ⦿ Catalysts
- ⦿ Paper pulp
- ⦿ MOFs

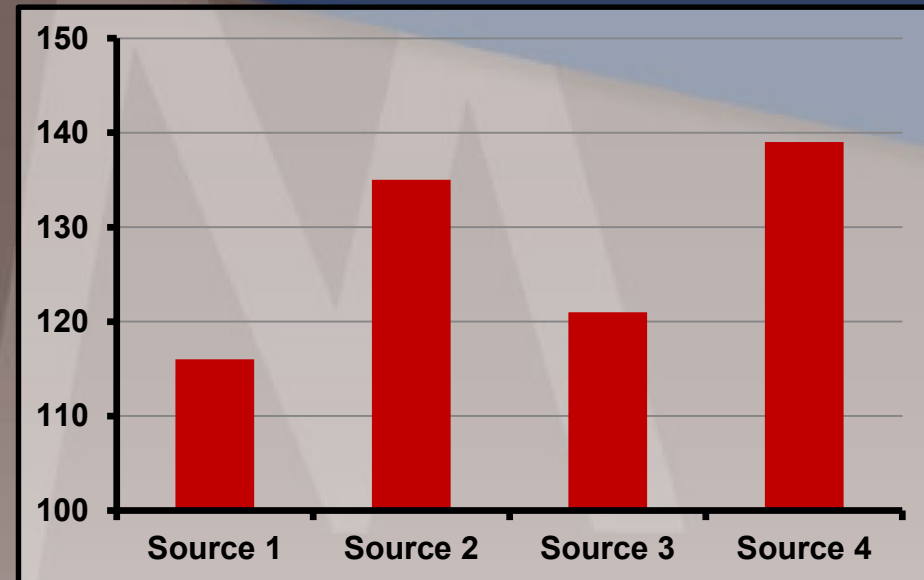
Incoming raw materials



The *MagnoMeter* is a fast, simple tool for easy comparison of raw materials



LIME: 11wt% in MeOH



ALUMINA: 37.5wt% in EtOH

Industrial raw materials are not pure → type and level of impurities depends on source and processing

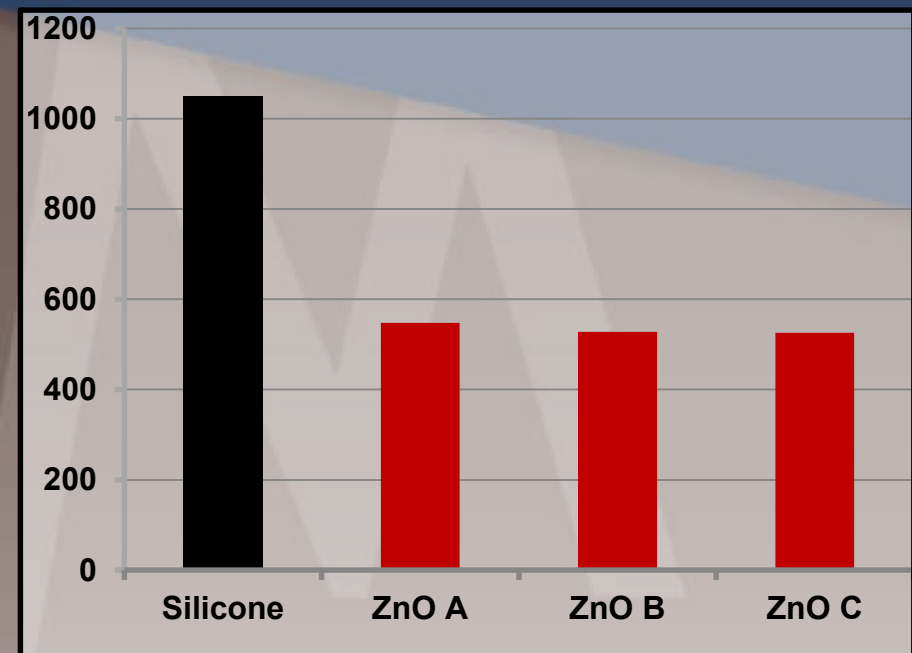
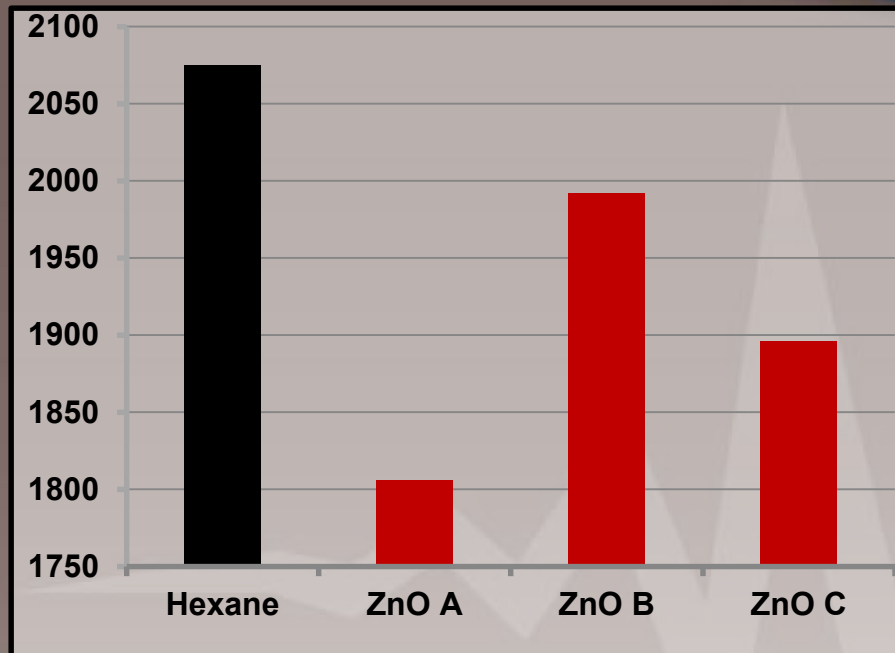
MagnoMeter measurements can be used as a quick estimation of product end-use performance testing

Type	NMR Surface Area* (m ² g ⁻¹)	Tensile Strength** (psi)	Relative Abrasion**	Relative Road Wear**
Oil Furnace	129	3600	1.35	1.25
Oil Furnace	90	3300	1.25	1.15
Gas furnace	44	2300	0.56	0.66
Gas furnace	34	1800	0.48	0.60

* 10 wt% Carbon Blacks dispersed in a mixed solvent

** SBR Rubber tires

The *MagnoMeter* provides fast determination of powder wettability



Average Relaxation Time repeatability: < 1.0%

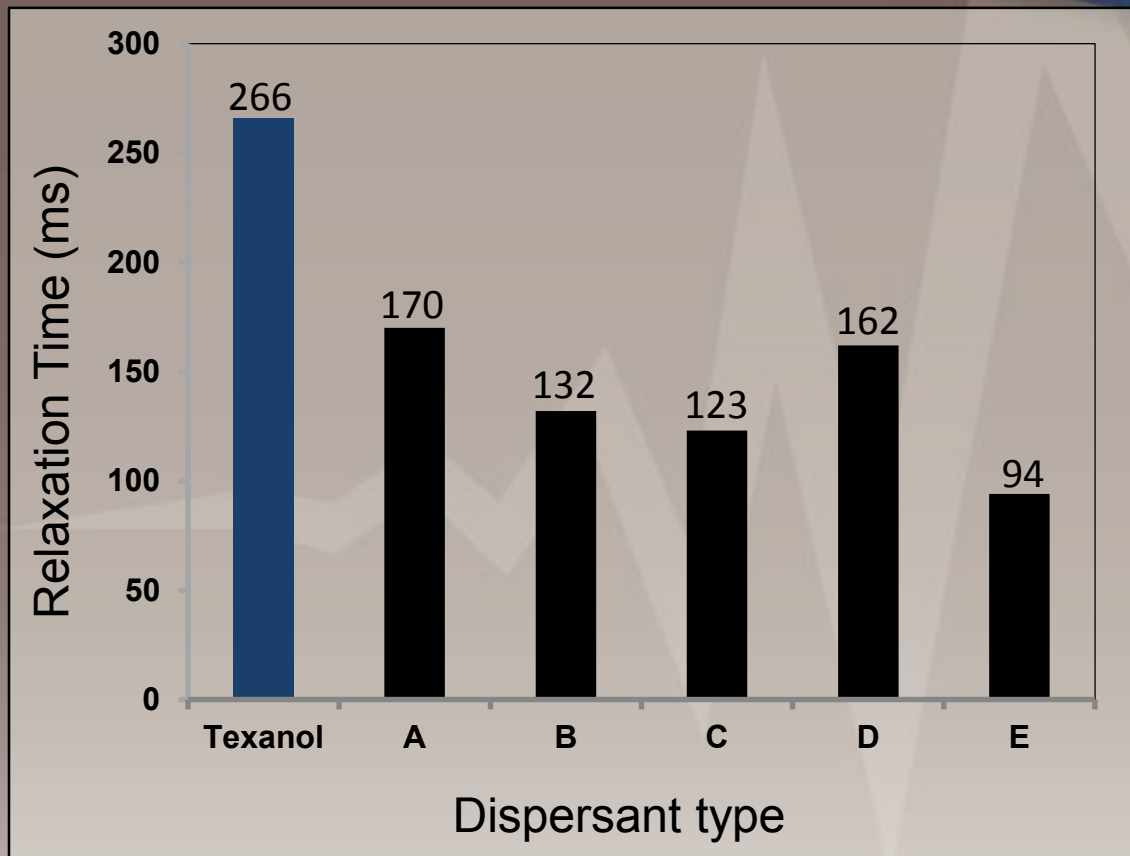
Comparison of relaxation time of a hydrophobic microfine ZnO* dispersed (8wt%) in hexane and 100 Cst silicone fluid

*Obtained from three different manufacturers (USA, EU and Japan) and claimed as equivalent products: same particle size and same silane-based "coating".

R&D: The Best Dispersant for a Silver Metal Powder?



The *MagnoMeter* allows rapid determination of the suitability and efficacy of dispersants



A: No dispersant
B: Zephyrym PD 2206
C: Hypermer B210
D: Crodafos M915A
E: Hypermer KD1

Average Relaxation Time
repeatability: < 1.0%

60wt% silver flake in texanol; 2wt% dispersant

R&D: Dispersion of a Blue Dye



The *MagnoMeter* is a fast tool to aid in optimizing the formulation and preparation of any suspension

Surfactant (trade name)	Surface Area (m ² g ⁻¹)
None	15.4
Fluorad	16.0
Natrol 42	18.5
Lomar D	35.5
Aerosol AOT	42.0
Ultravon W	46.2

Increasing
Opacity

Increasing Color
Intensity

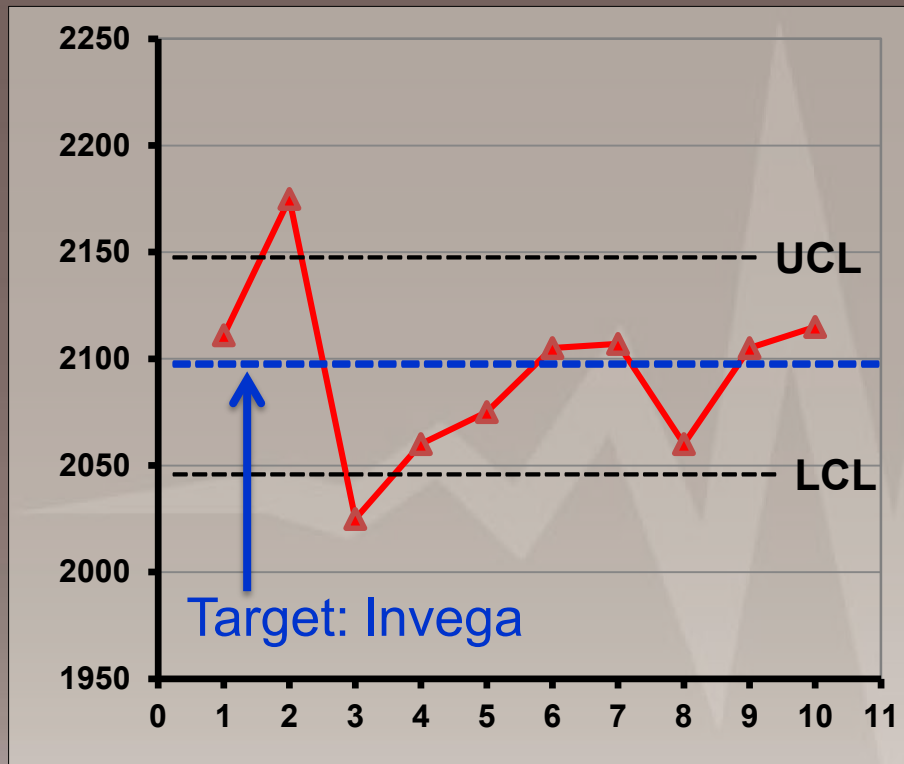


Aqueous blue (carboxy) dye suspension: 20% wt/wt; pH10

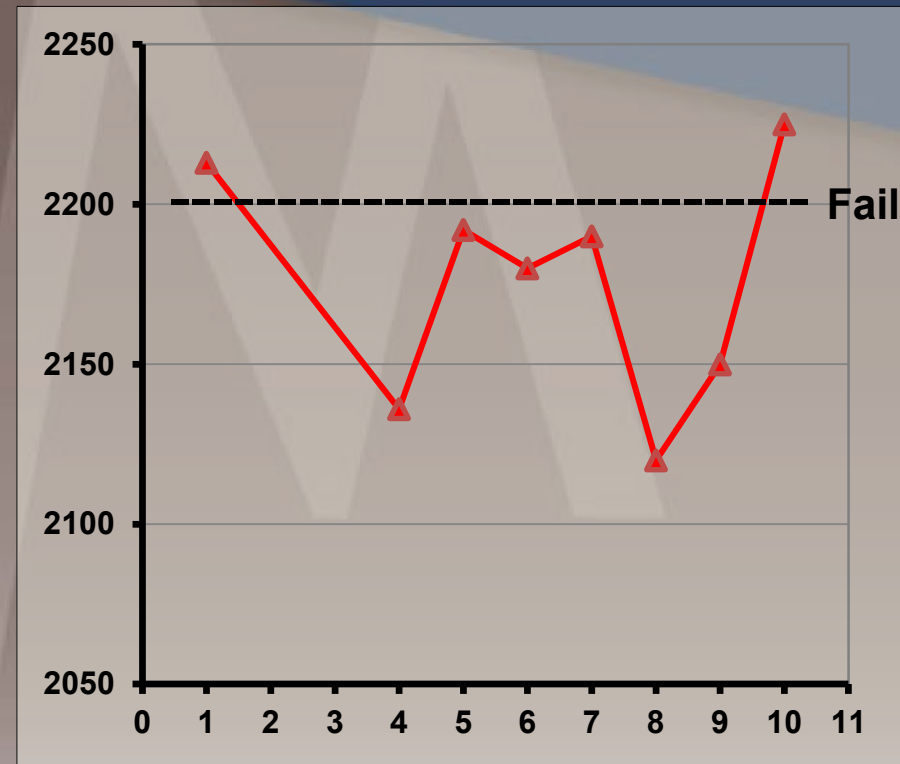
QC: An API Product



MagnoMeter measurements are non-destructive
samples can be stored and re-analyzed



Relaxation time of batches
of paliperidone palmitate



Pass batches after 3 months
aging at 40°C

QC: Ink-Jet Pigments



MagnoMeter measurements are very fast
allowing for rapid QC of dispersions

Pigment	Solids Wt%	Surface Area (m ² /gm)	Pass/Fail
Y1A	12.9	83	Y
Y1B	13.4	60	N
Y4C	10.8	30	Y
Y4D	10.0	26	N
M1A	18.3	90	Y
M1B	18.5	31	N
C1E	15.6	56	Y
C1F	15.3	46	N

Aqueous suspensions of Yellow, Magenta and Cyan Pigments

QA: Lotions

MagnoMeter measurements can be used to rapidly screen commercial products

Comparison of six commercial Lotions

Product*	Cost (\$/kg)	Short T ₂ (ms)	Oil Phase %	Long T ₂ (ms)	Water Phase %
Jergens	57	278.1	30.0	540.7	70.0
Generic	16	80.2	21.3	301.7	78.7
Aveeno	51	147.7	53.1	337.1	46.9
Generic	38	255.7	43.3	497.0	56.7
Gold Bond	26	47.1	24.0	244.1	76.0
Generic	21	76.9	47.6	251.0	52.4



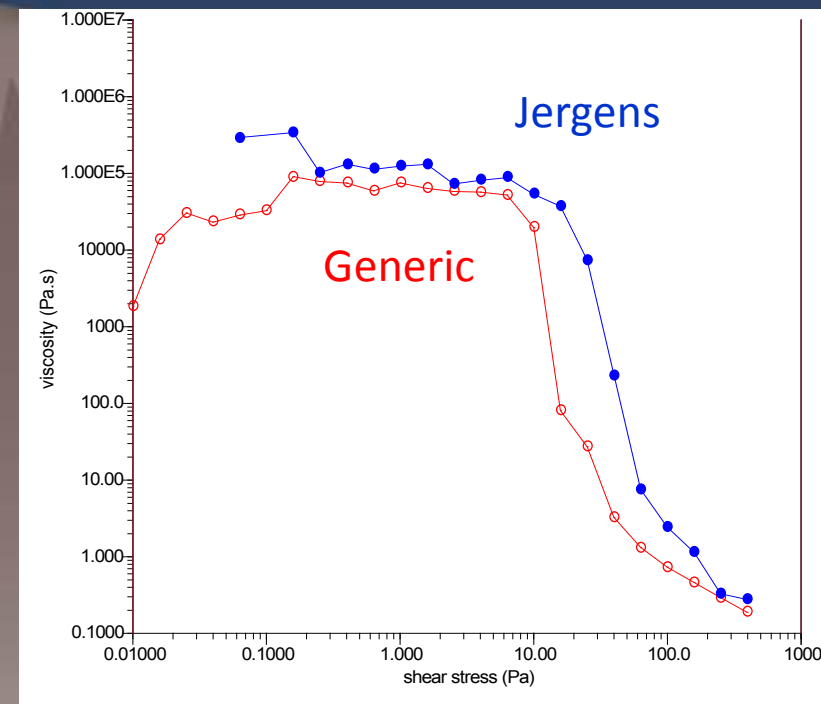
** Composition of each product pair is similar
Data suggests that product performance behavior will differ

Lotions: Rheology data*



MagnoMeter measurements correlate well with emulsion visco-elastic characteristics

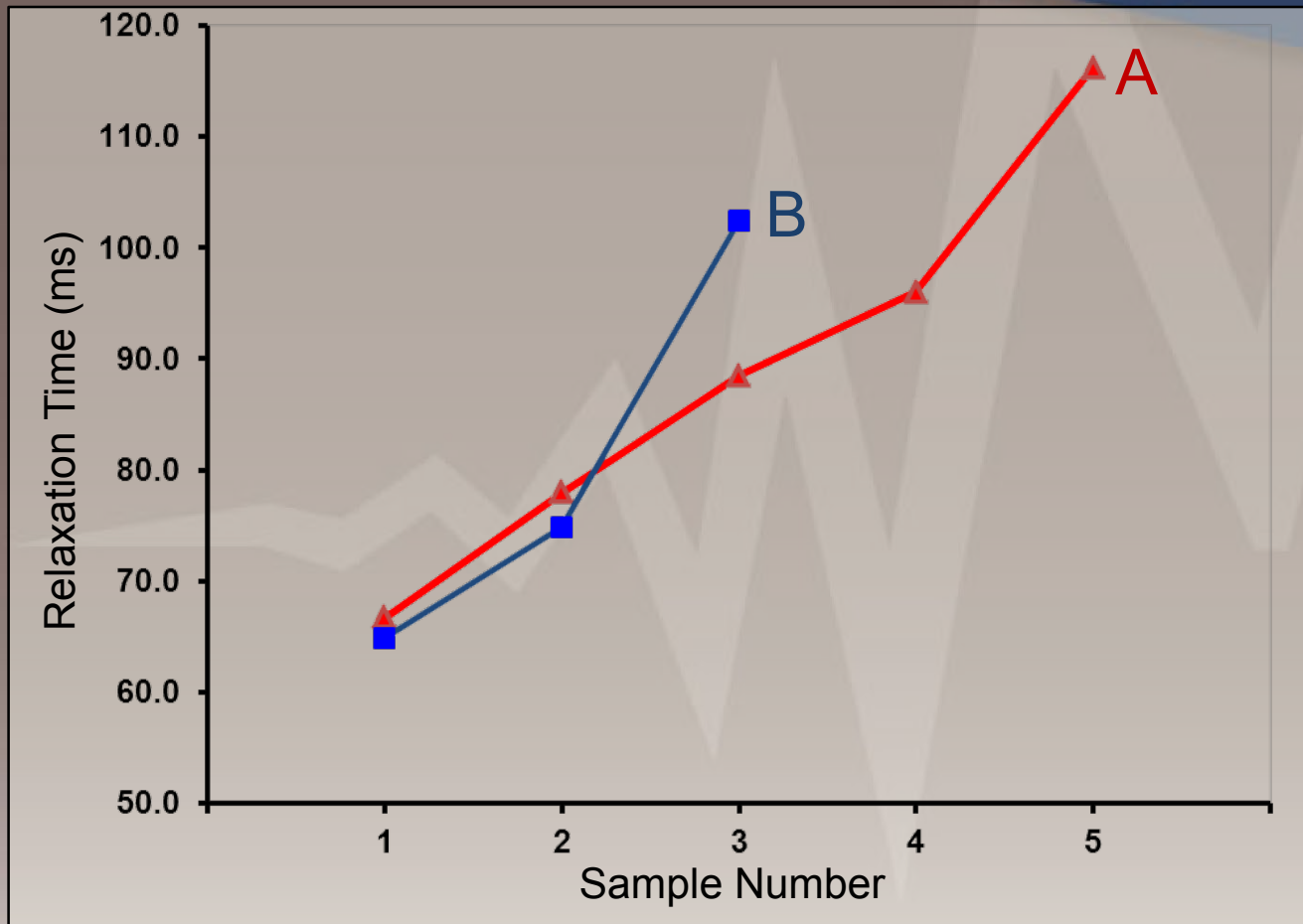
Product	Viscosity (Pa.s)	Yield (Pa)	Short T_2 (ms)	Long T_2 (ms)
Jergens	93,640	20	80	302
Generic	66,790	10	278	541
Aveeno	280,170	100	256	497
Generic	450,040	170	148	337
Gold Bond	233,700	60	77	251
Generic	280,400	110	47	244



For each emulsion pair: (a) as Viscosity increases, the *long* T_2 is seen to decrease
(b) as Yield increases, the *short* T_2 is seen to decrease

Processing: Graphene Oxide

MagnoMeter measurements are very fast, and processing can be followed in almost real-time



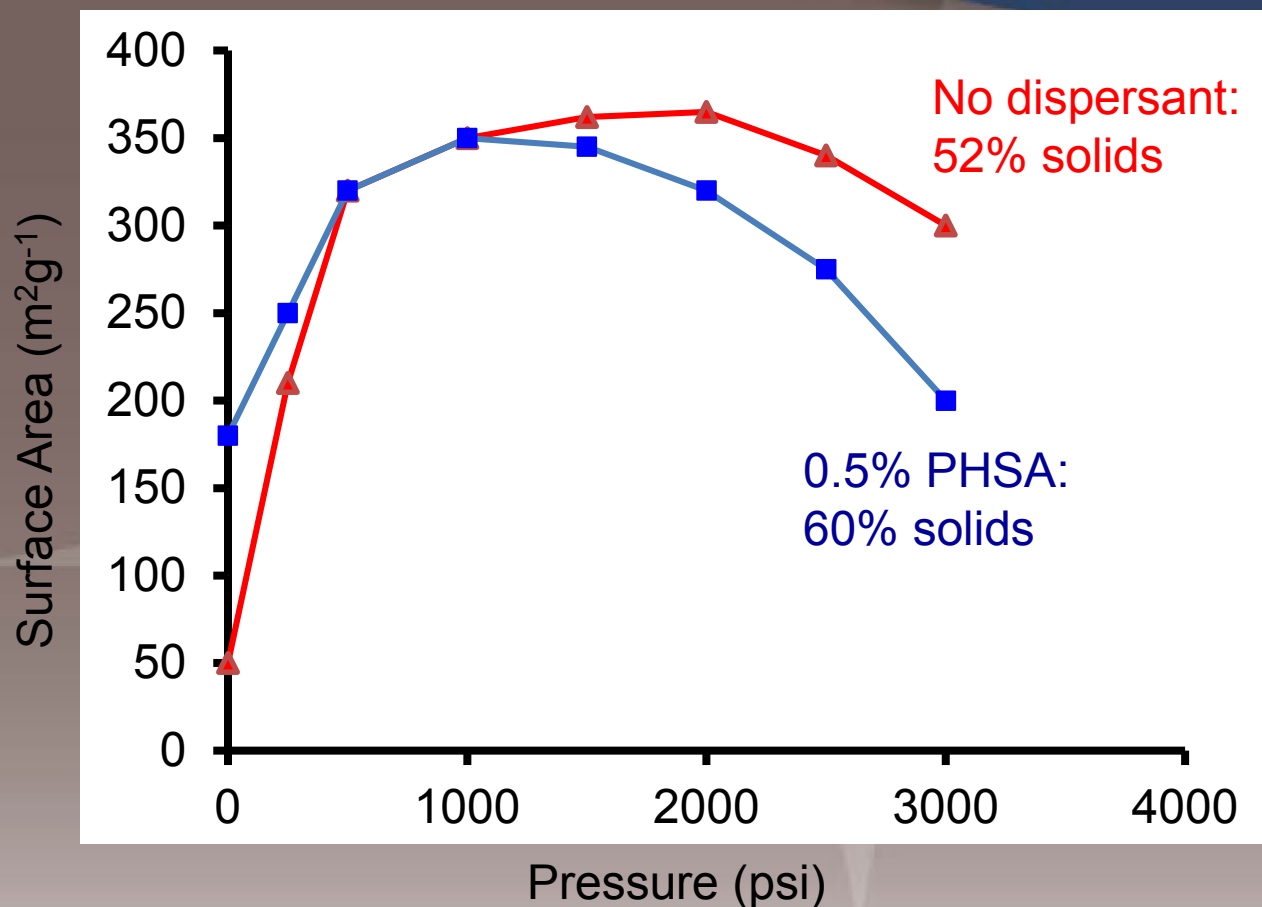
Two sets of GO
process material:
slurries in water
at 3wt%

Average Relaxation
Time repeatability:
< 1.0%

Processing: Zinc Oxide



MagnoMeter measurements do not require dilution – samples can be taken directly from mill for analysis

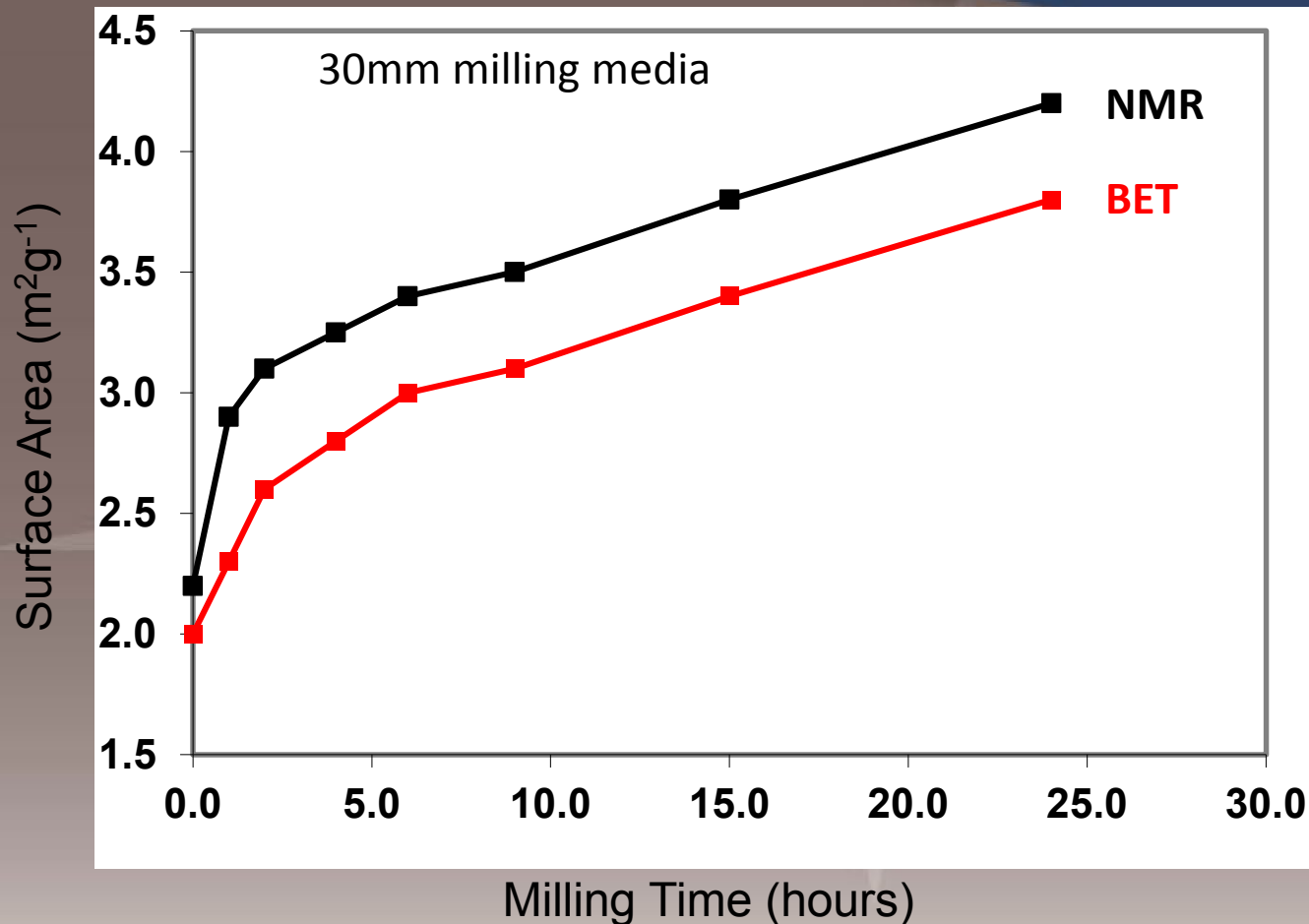


Two sets of ZnO
process material:
slurries in C₁₂-C₁₄
alkyl benzoate

Processing: Whitewares



Direct *MagnoMeter* measurements on 30% ceramic millbase slurry trends with BET (gas adsorption) data



Magnometer
Measurements
take minutes, not
hours – and with
no sample
preparation

What does the *MagnoMeter* provide?

- ⌘ Direct information about the extent and nature of *any* particle-liquid interface → suspensions and emulsions
 - ⌘ any type of particle, and any liquid – including mixtures
 - ⌘ exceptionally wide concentration (0.01% to 90+%)
 - ⌘ no dilution required, minimal sample preparation
 - ⌘ small samples (typically 0.1mL; as little as 200μL)
- ⌘ Complementary information to traditional particle characterization techniques
- ⌘ Intelligence that is not possible with traditional techniques

Advantages of the *MagnoMeter*

General

- ⊗ Any colloidal-size suspension – virtually all liquids
- ⊗ Measure locally with data manipulation at remote terminal
 - ⊗ Ideal for controlled or hazardous environments
- ⊗ Multiple pod sensors facilitate sampling at different locations
- ⊗ Frequency lock
- ⊗ Measure mixture homogeneity

Options and Extensions

- ⊗ Large diameter NMR tube
 - ⊗ viscous liquids, concentrated slurries/emulsions
- ⊗ MRI methods (imaging) in specific samples
- ⊗ Can be adapted for use with an auto-sampler
 - ⊗ multiple sample analysis
- ⊗ Can be adapted for flow-through operation

The *MagnoMeter* finds use from Fundamentals to End-Use Performance

✿ *Research & Development*

- ✿ Basic formulation of products: reproducibility, stability (coagulation/flocculation), settling and sedimentation, shelf life

✿ *Quality Control*

- ✿ Release of raw materials, batch-to-batch reproducibility of final product

✿ *Quality Assurance*

- ✿ Enable release of complex systems which can currently only be characterized by the raw materials used rather than the manufactured product

✿ *Process Management*

- ✿ Follow and monitor milling and manufacture processes in almost real-time

THANK YOU!

For more information, to send samples, to arrange a demonstration at your facility, or to speak to a technical applications specialist, please contact:

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