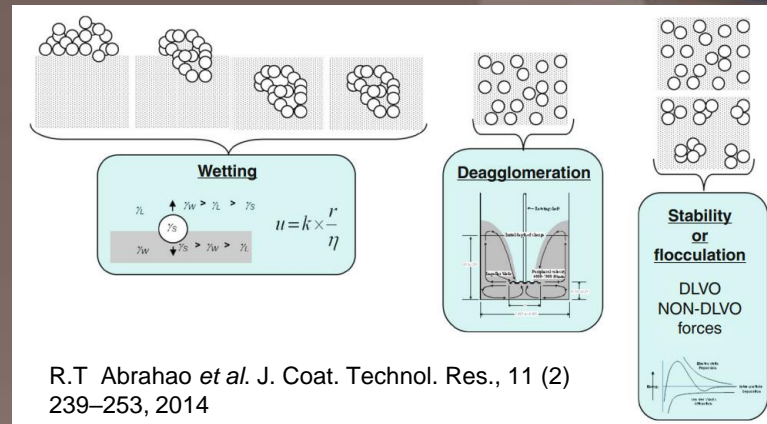


The Use of HSP in Formulation Development



Coatings, inks, pharmaceuticals and cosmetics etc., increasingly employ micro- or nano-particles carefully formulated in a variety of carrier fluids



- Initially dispersing a powder into a liquid phase is a critical process step in formulating and manufacturing
- A predictive method for selecting an appropriate solvent (or solvent mixture) in wetting and dispersion of powders has practical and economic benefits
- Hansen Solubility Parameter (HSP) method is a useful approach to predict solvent quality for wetting of powders**

HSP applied to dispersion of particles

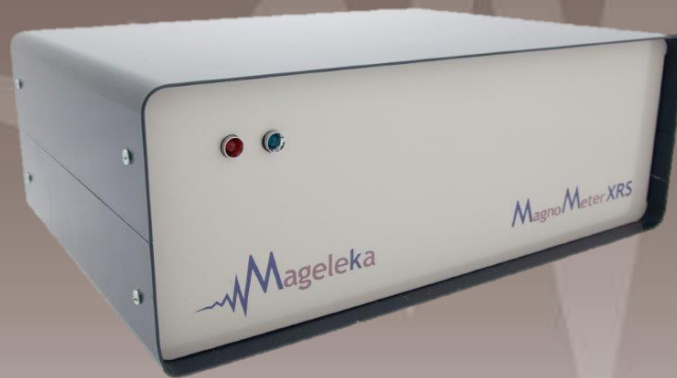


- ⊗ Sedimentation time used as suitable metric to determine HSP
 - ⊗ Traditionally measured simply by eye → settling slowest in good solvent
 - ⊗ very subjective;
 - ⊗ very time-consuming for nanoparticles;
 - ⊗ no standardized procedure
 - ⊗ Analytical centrifugation (AC) – major advance
 - ⊗ Significantly faster; provides quantification of particle agglomeration
 - ⊗ Rank order of solvents
 - ⊗ SOP developed to quantitatively determine HSP of a solid material
 - ⊗ Limitations of the AC method
 - ⊗ Spherical particles
 - ⊗ Narrow size distribution
 - ⊗ Low concentrations
 - ⊗ Needs corrections
 - ⊗ Time consuming

Mageleka's *MagnoMeter*



- ⊗ Fast (2 min), direct and simple measurement
- ⊗ Size and shape of material not relevant
- ⊗ Any Industry relevant solids concentration
- ⊗ No corrections



Examples



Zinc Oxide, ZnO

Property	Coating	Nature *	Zeta potential** (mV)	Mean Particle Size (nm)
Hydrophilic	None	Cationic	+39	ca 120
Hydrophilic	SiO ₂	Anionic	-55	ca 160
Hydrophobic	Silane	Non-wetting	N/A	ca 140

Alumina, Al₂O₃

Property	Coating	Nature*	Zeta Potential (mV)	Mean Particle Size (nm)
Hydrophilic	None	Cationic	+45	ca 300
Hydrophobic	Silane	Non-wetting	N/A	ca 300

* In water; ** In 10mM KCl (aq)

Zinc Oxide, ZnO

Selected from**:

Acetone, Acetonitrile, Benzyl Alcohol, Benzyl Benzoate, Butanol, Caprolactone, Chloroform, Decyl Alcohol, Dichloromethane, Dimethylformamide, Dimethyl Sulfoxide, Dodecane, Ethanol, Ethyl Acetate, Ethyl Lactate, Ethyl Oleate, Heptane, Hexane, Isopropanol, Methanol, Methyl Cellosolve, Methyl Ethyl Ketone, Methylene Chloride, N-Methyl Pyrrolidone, Propylene Carbonate, Tetrahydrofuran, Toluene

Alumina, Al₂O₃

Selected from above plus:

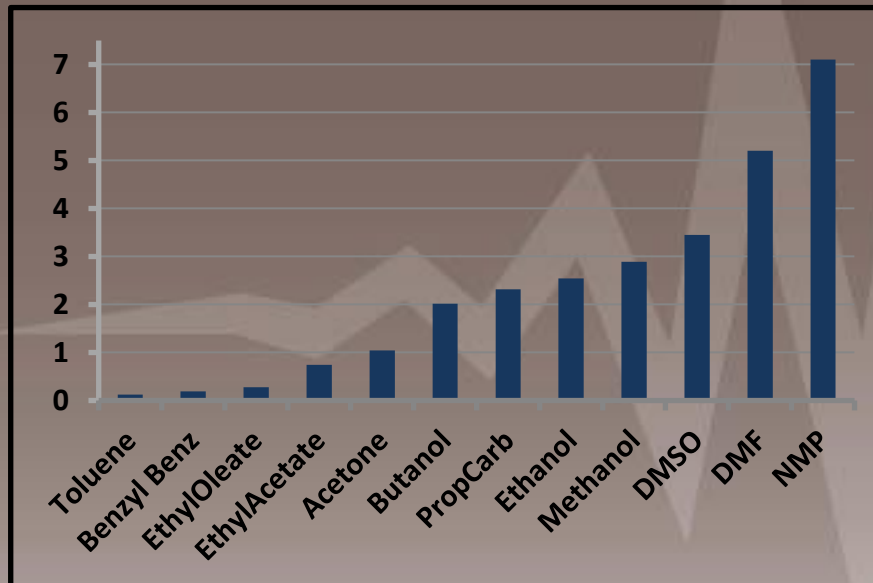
Cyclohexane, Cyclopentanone, Diacetone Alcohol, Dioxane, N-Methyl Formamide

Experimental Results: ZnO

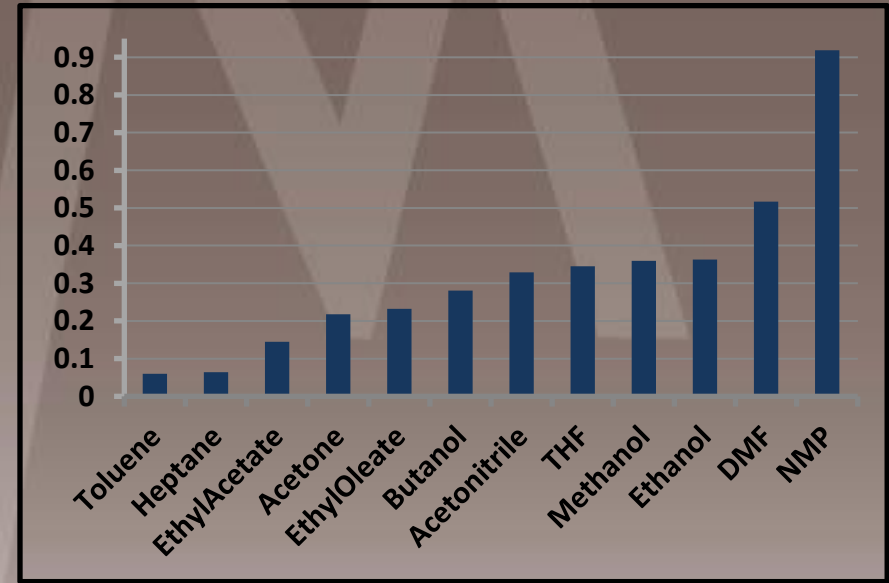
Relative Relaxation Rate, R_{sp} , for two Zinc Oxide powders are significantly different depending on solvent-surface interaction

$$R_{sp} = [R_{susp}/R_{solv}] - 1$$

Silica coated



Silane coated

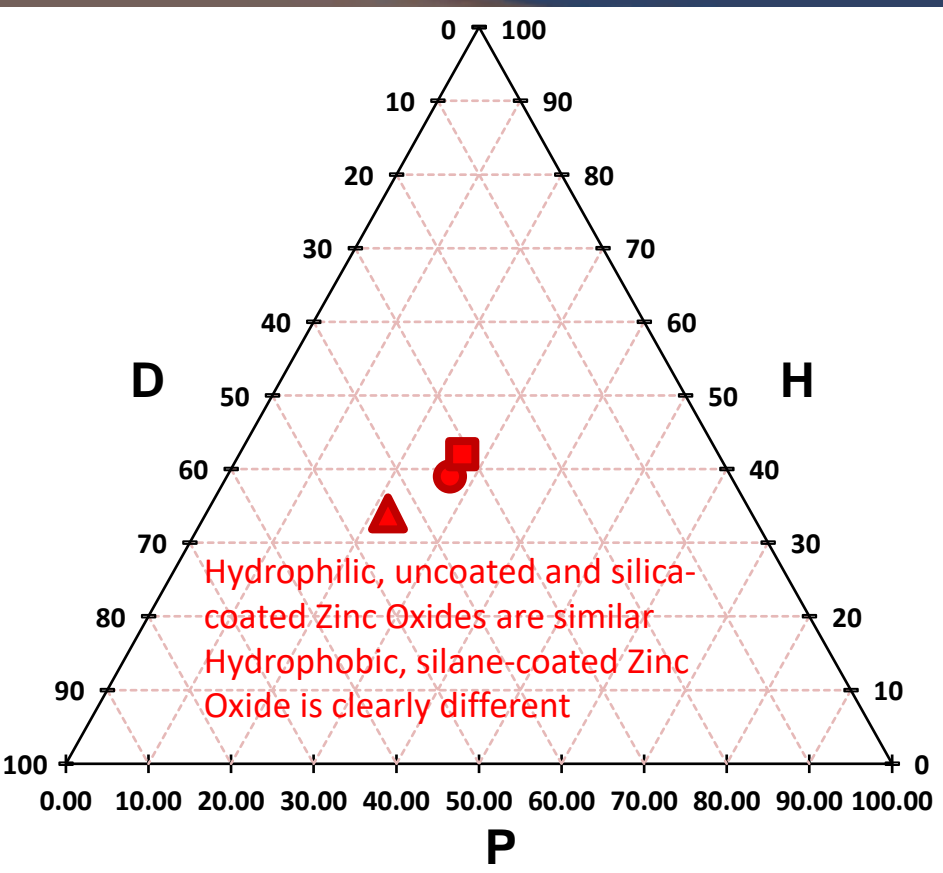


More efficient wetting \rightarrow larger R_{sp} value

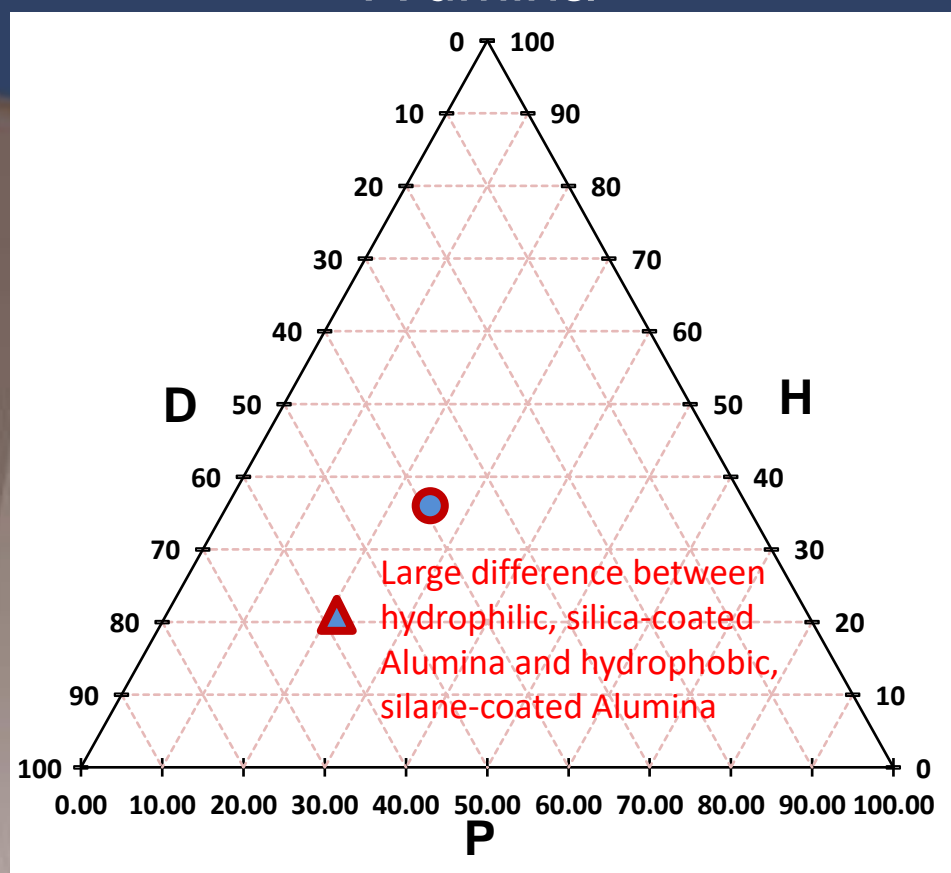
TEAS Plots: Comparing Hydrophilic ZnO and Al₂O₃ vs their hydrophobic derivatives



Zinc Oxide



Alumina



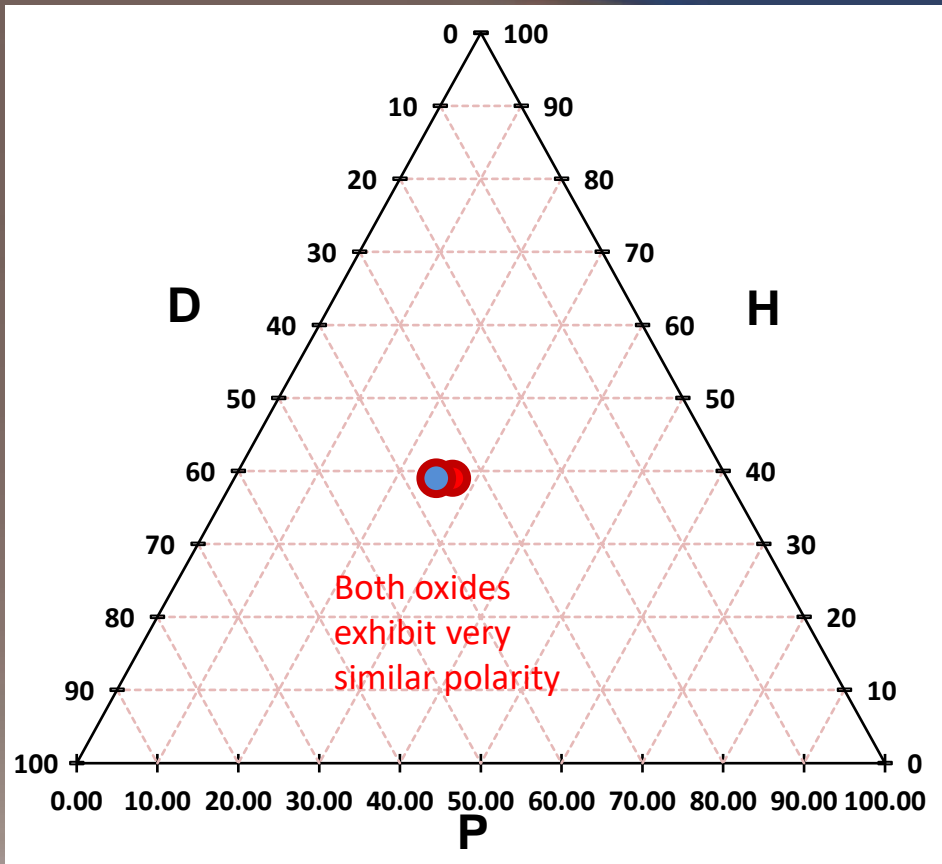
Uncoated ● Silica coated ■
Silane coated ▲

Uncoated ○ Silane coated ▲

Comparing ZnO and Al₂O₃ and their hydrophobic derivatives

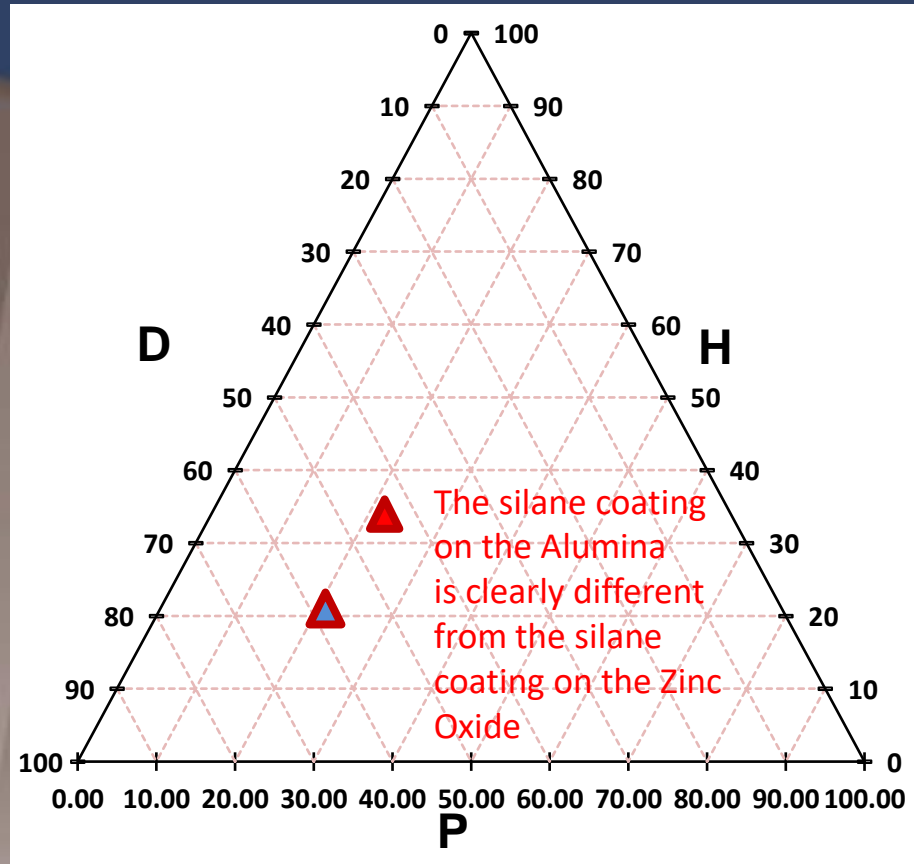


ZnO/Al₂O₃ Uncoated



Zinc Oxide ● Alumina ○

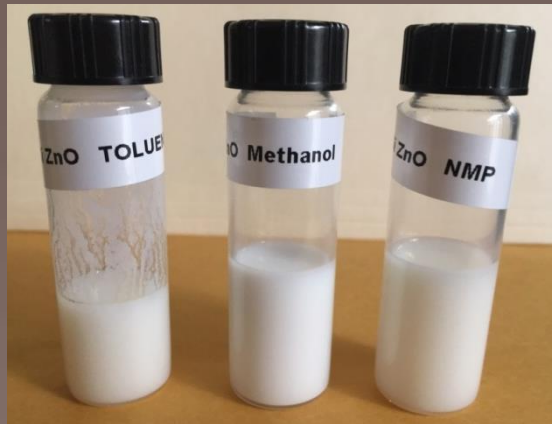
ZnO/Al₂O₃ Silane coated



Zinc Oxide ▲ Alumina ▲

Silica-coated Zinc Oxide dispersed in three different solvents

(a) After initial sonication



(b) After 4 hours



Poor wetting of the glass vial by the Toluene suspension; **Methanol and NMP suspensions both look good**

MagnoMeter results show clear difference
NMP (7.10) > MeOH (2.89) > Toluene (0.12)

Toluene is very poor wetting agent for the zinc oxide powder.; NMP is most efficient

Toluene suspension: separated and flocculated.

Methanol suspension: noticeable sediment

NMP suspension: virtually no sediment

MeOH able to wet the powder but is a less efficient dispersant

Conclusion



The *MagnoMeter* is a powerful tool for selecting suitable solvents for wetting and dispersion of powders

Measurements can

- ⊗ discriminate between surface chemical coatings
- ⊗ distinguish between suspensions that visually look, initially, to be similar
- ⊗ provide time-saving information in formulation

MagnoMeter measurements provide fast and simple means to determine the HSP of solid materials*

* D. Fairhurst, R. Sharma, S. Takeda and S. Prescott, *Powder Technology*, 377 545-552 (2021)