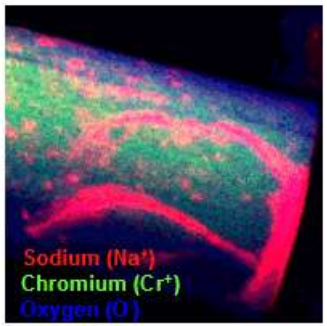

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Technical Industrial Problem Solving and Failure Investigation at LPD Lab Services

Dr Stephen Jenkins – Managing Director / Principal Scientist

MEDIPLAS 2013 (NEC) - 26th September RAPRA Session Invited Speaker

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LPD Lab Services

Who are LPD Lab Services?

- Mix of analytical industrial chemists, materials scientists / engineers, physicists (Over 180 man-years experience – 8 technical staff)
- Access to Consultants and Trusted Partner Laboratories
- Diverse product and process manufacturing knowledge.
- Experienced and pragmatic problem solvers backed by 6 Sigma expertise.
- Used to providing quick and effective solutions to deal with unusual problems
- Diverse laboratory equipment – right tools for job!
- Skilled in bespoke sample preparation without interfering with physical and chemical structures.
- Flexible / proactive approach to scope of work.

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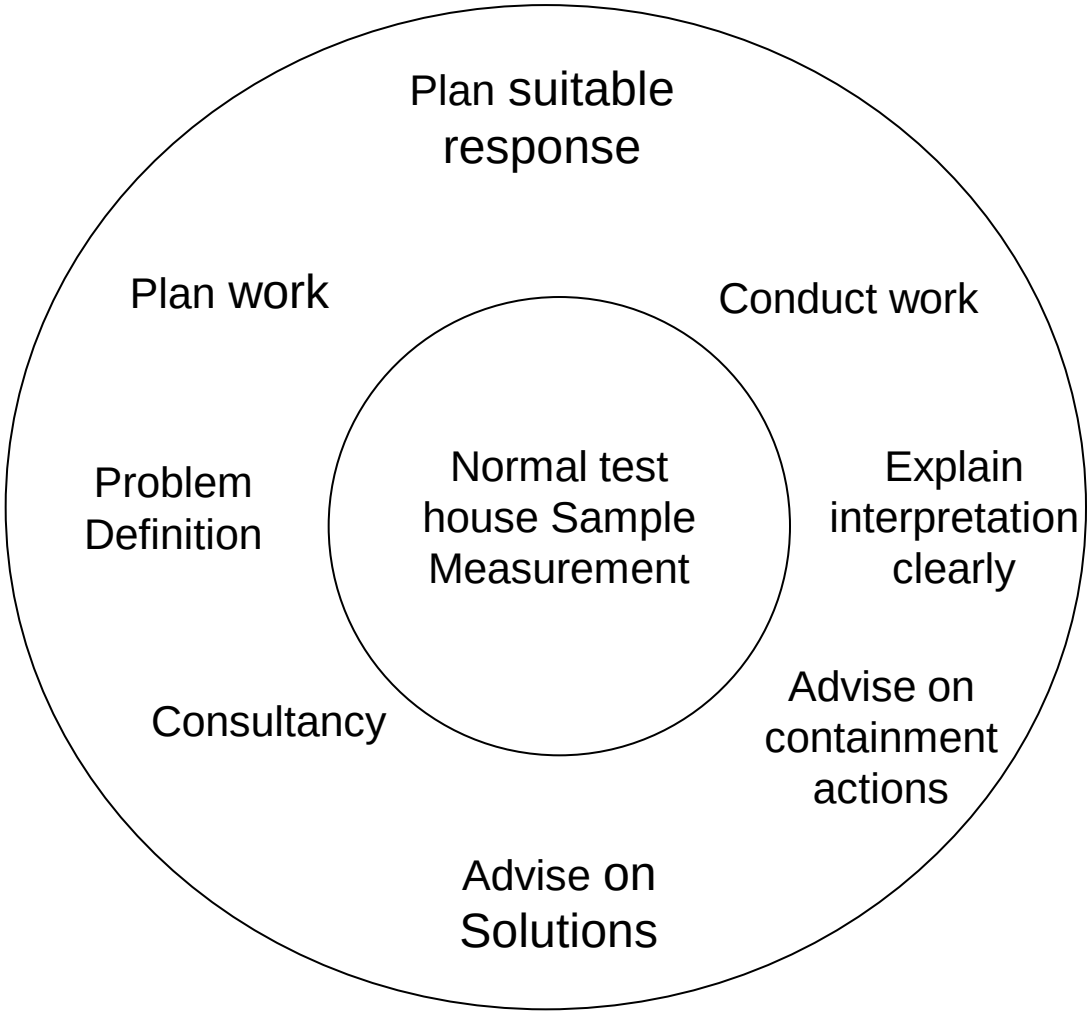
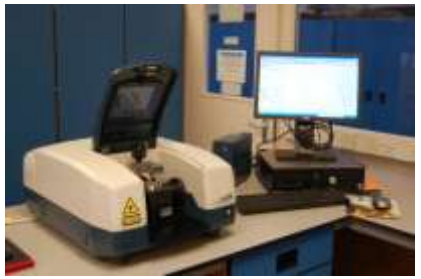
Problem Solving Approach – Beyond a Test House





Problem Solving Methodology:-

- 6 Sigma
- 8D
- Kepner Tregoe (KT)

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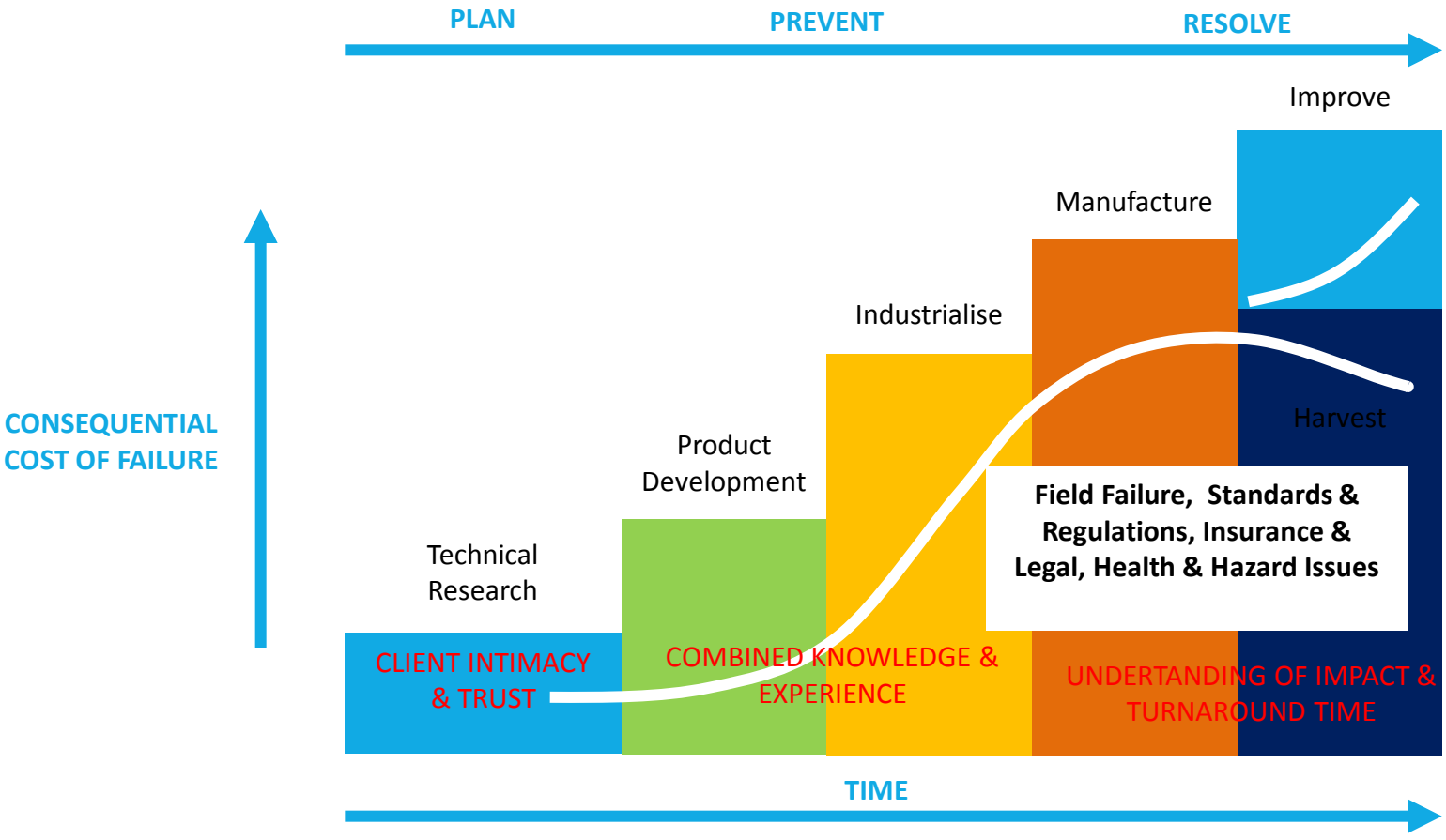
Support Service Life Cycle



Introduction to Specialities of LPD Lab Services

Specialties:-

- Physical Analysis
- Chemicals Analysis.
- Materials analysis and materials engineering
- Surface analysis
- Bespoke tests and measurements
- Problem Solving
- Consultancy
- Reverse Engineering / Deformulation
- Product and process development.

Key Factors –

Competent, Experienced, Fast, Adaptable and communicative

Turn **complex data** into **understandable practical information.**

Accreditations –

ISO 17025:2005 (laboratory)

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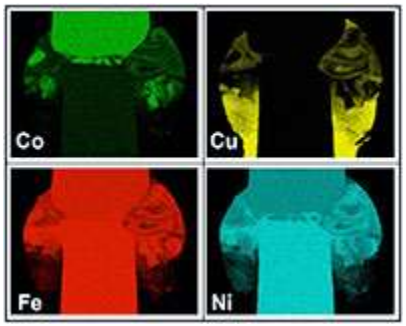
5

Materials Analysis

Why?

- Generate improved performance and quality of products
- Cost reduction
- Identify, track and remove contaminants



Involves:-

- Materials Analysis and Failure Investigation
- Physical, Structural and Microstructure Analysis
- Chemical Analysis.

Materials analysis instrumentation:-.

- **Optical Microscopy, SEM/EDX, FTIR, XPS, SIMS, XRF, AAS**

Cost and Time efficiency:-

- Failure analysis allows skillful dismantling products and components to solve problems - **Determine material and product shortcomings.**
- Reverse engineering **benchmarking of competitors** products to reveal the production methods and materials - **Drive product development.**

LPD Lab Services

Physical, Structural and Microstructural Analysis

Physical properties, mechanical properties, structure and microstructure of:-

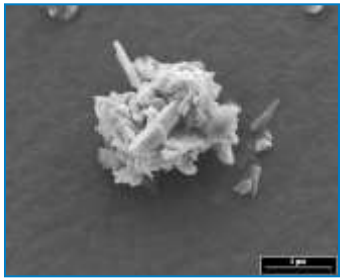
- **Chemicals**
- **Materials**
- **Assembled products.**

Including:

- Top down inspection – Optical Microscopy and SEM
- Cross-sectional analysis – Optical Microscopy and SEM
- Image analysis

- Chemical compatibility and wetting (contact angles) - DCAT/OCAT
- Mechanical Tests and Hardness

- Viscosity
- Particle size, size distribution and shape.



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Chemical Analysis

- **Gases, Liquid** or **Solids**
- Bespoke **Method Development or Routine analysis**
- Wide **array of analytical techniques**:-
  - FTIR, XRF, AAS, ICP-MS, GC, HPLC, IC, UV/Vis, Karl Fischer, Titration, Back extraction
- Practical **Context-orientated interpretation**

Example work:-

- Product / process control and validation
- Identification and elimination of contaminants
- Chemical Product Deformulation and Reformulation
- Adhesives, coatings, adhesion promoters and coupling agents
- Analysis of binders and fillers
- Determination of plastic / polymer types
- Identification of unknown chemicals and detergents
- Trace metal analysis

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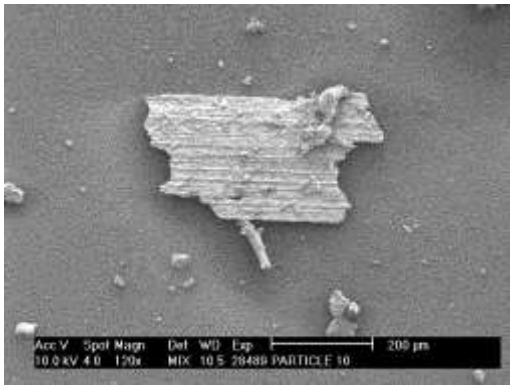
Particle Contamination Identification and Elimination

- **Particle contamination is problematic for many industries.**
- **Isolating and characterising the particles (with Optical Microscopy, SEM / EDX, FTIR and SIMS) can yield a likely source.**

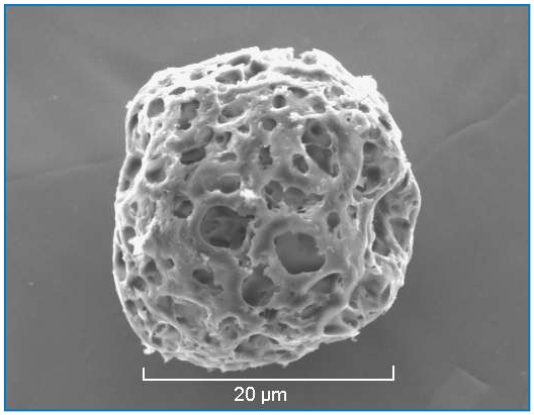
Perform this work on virtually any type of sample, including:

- Liquid samples – suspended particles (pharmaceuticals).
- Filters / Membranes (eg inhaled products).
- Product surfaces and under transparent polymer layers eg catheters, other medical devices.



Metal oxide particle with characteristic machine marks

Charred pollen grain found to block a filter.



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Materials Problems with Plastics and Coatings?

Plastic Moulding Cracking Over Life

Moulding conditions can leave residual stress
Relieved over life giving cracking (Environmental Stress Cracking - ESC)

- Solvent or cleaning agents give polymer chains mobility – Cracks.
- Cracks trap bacterial and hard to clean.
- Affected by filler contents, filler types, size shape and distributions
- Polymer crystallinity.

Shrinkage

- Mobile species like plasticisers or low molecular weight polymer can leach out under heat.
- Change mechanical properties.
- Cause delamination.

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Materials Problems with Plastics and Coatings?

Delamination

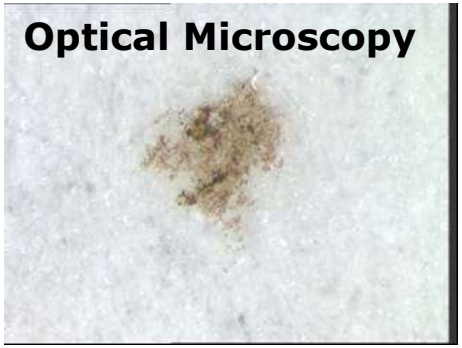
- Incomplete Curing
- Weak boundary layers

Depolymerisation / Degradation

- Stabiliser concentration problems – Material less robust.
- Hydrolysis.
- Oxidation.
- Free radical attack.
- Attack by mobile metallic ions.
- Chlorine embrittllement in Polyacetal.

Buried Particles in Wound Dressing Laminate

Particle identification, isolation, characterisation and type matching in medical laminate.



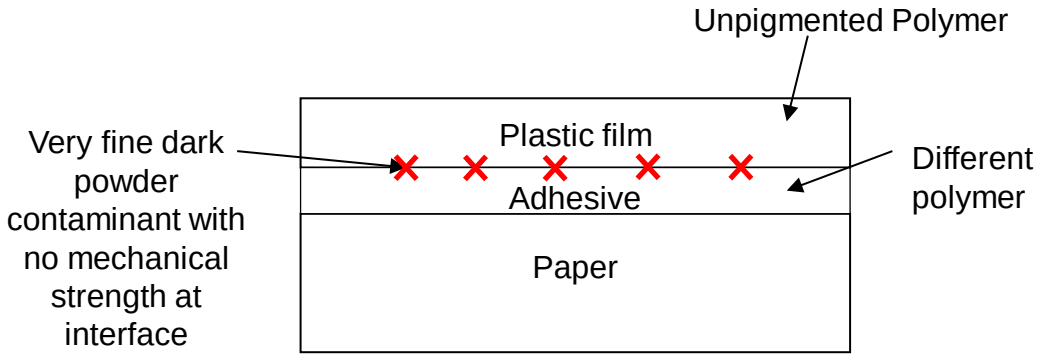
150µm diameter particle

- Dismantle laminate under microscope

OR

- Solvent removal of over-layers without disturbing physical and chemical structure so causal link can be proved

SEM/EDX showed particles in raw and finished laminate materials chemically and physically similar (iron oxide + other elements) to confirm supplier was source.



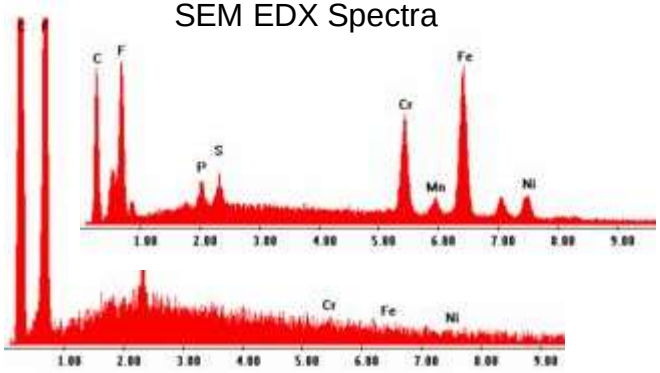
Identification of Sources of Particles in Products

Example particles found in products:-

Wear Particles

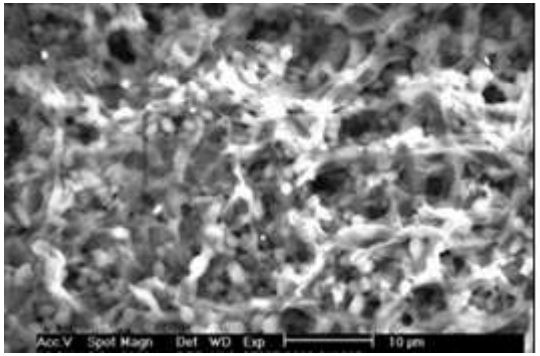
- Broken Fluorinated rubber with embedded stainless steel from mixer seal – Wear material.
- Fine structure and density points to source.

SEM EDX Spectra



Algae

- Typical nodular structure, density and chemistry (FTIR & SEM/EDX) of algae.
- Low Magnification images showed flat sides indicated dislodged from sidewalls of pipe or process tank.



Algae SEM Image

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Materials for Wound Management & Infection Control

- Physical and Chemical Interactions between Silver Alginate wound dressing materials.
- SEM to look at fibre types and different fibre fractions / distributions
- XPS to investigate residual surface treatments from processing slip agents or treatments to affect antibacterial activity.
- Process control checking CHDG (Chlorohexadine Digluconate) in antiseptic wipes.
- Development of staining systems to visualise active ingredient distribution like CHDG in fabrics (wipes and dressings) in cross-sections.

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Summary and Final Comments

RAPRA
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Effective Problem Solving Needs…

- Experienced analytical staff to recognise failure mechanisms.
- Combine interpretation from multiple techniques.
- Act as extra R&D manpower / consultants for customer.
- Actively input into product and process modifications.
- Add value input when analysts have close understanding of the product and design requirements.
- Adaptable and react fast.

Any Questions?

LPD Lab Services Ltd

www.lpdlabservices.co.uk
enquiries@lpdlabservices.co.uk
Tel. 01254-676074