A Statistical Approach to Ink Formulation

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LEE-BED is a Horizon 2020 project that aims to reduce development time of materials and process for the production of hybrid electronics. CPI-Formulation's focus is on ink, adhesive and composite formulation, while CPI-Electronics are optimising testing procedures for roll-2-roll printed components. DTI is working on scaling up the synthesis of a range of nanoparticles, including copper nanoparticles. Other partners include Fraunhofer, TNO, ITENE, RISE, VSParticles, TPU, Swarovski, Maier, Acceona and Graphetic.

Experimental Planning

Copper nanoparticles were produced by DTI for LEE-BED with the aim of formulating an inkjet ink. The formulation already contained binder and other additives, however the drying properties needed to be optimised by using combinations of solvents.

Twelve polar mainly alcohol-based solvents were selected to be trialled. However testing all 12 solvents would result in hundreds of formulation iterations, therefore principle component analysis (PCA) was used to reduce the number of solvents investigated.

PCA is statistical way to group materials by their properties to identify similarities. Selected properties included boiling point, vapour pressure, surface tension, viscosity, density, flash point and Hansen solubility parameters. This identified the full parameter space from which five solvents which were selected; isopropyl alcohol (IPA), propylene glycol (PG), diethylene glycol monoethyl ether (DGME), benzyl acetate (BA) and dipropylene glycol propyl ether (DPGPE).

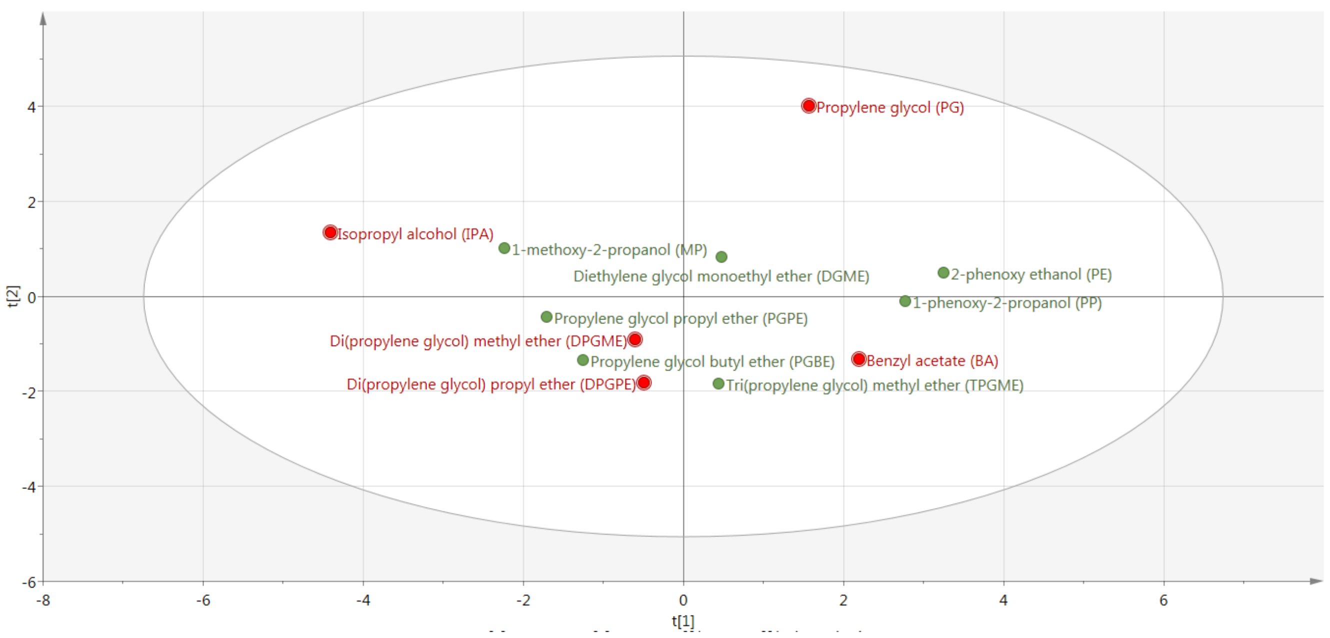

Figure 1 – PCA analysis of solvents; five were selected from across the parameter space

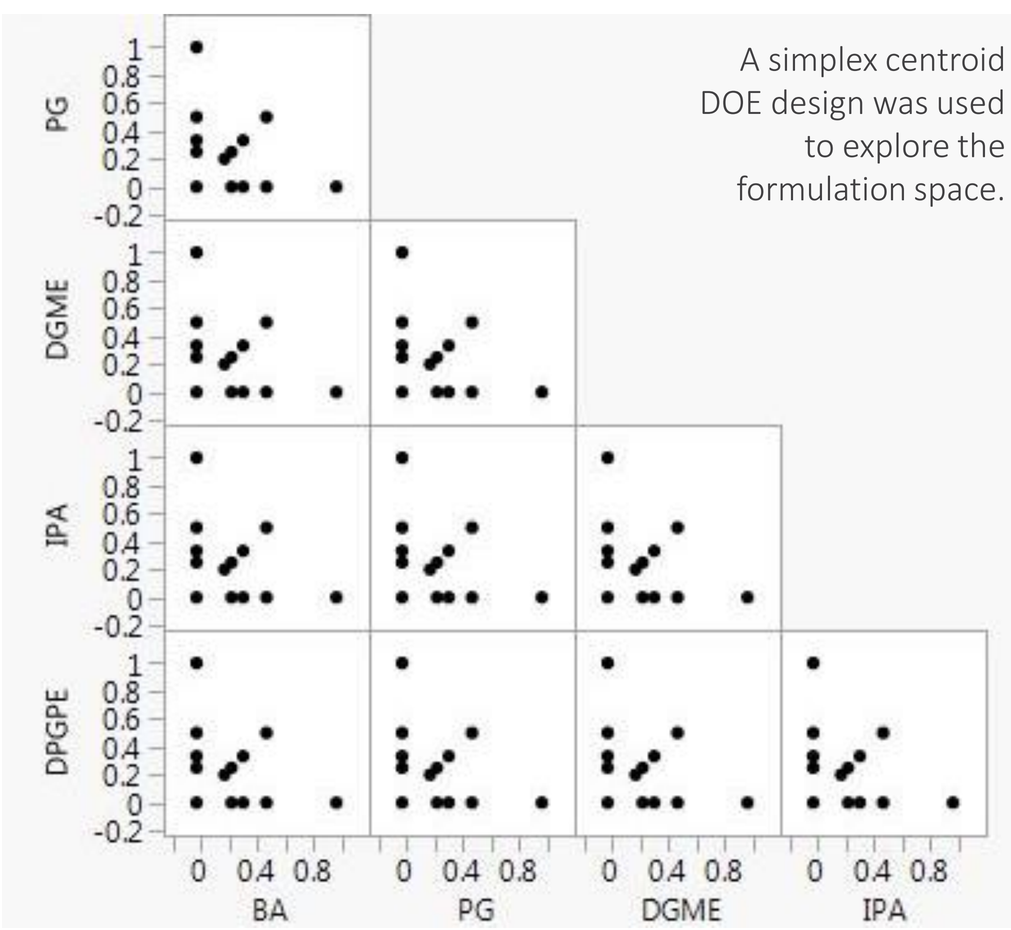

Figure 2 – Description of formulation iterations; each solvent individually, mixed 1:1, mixed 1:1:1 etc.

Formulation Procedure

Samples were made using a Chemspeed FORMAX robot, fitted with formulation vessels containing a rotor stator. Binder was added to the solvents and other additives while mixing.

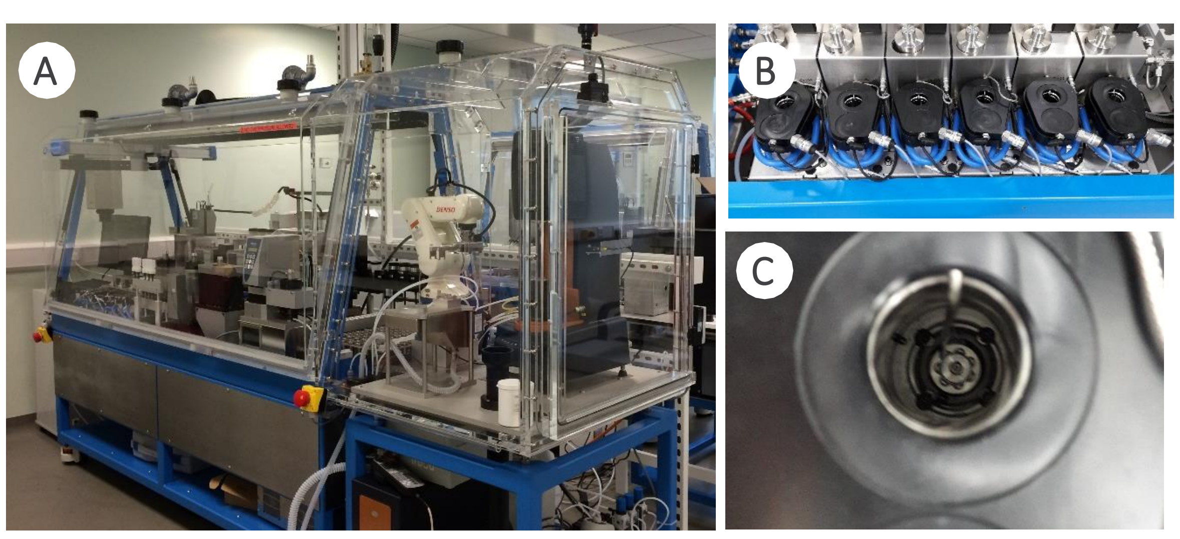

Figure 3 (A) – Chemspeed FORMAX formulation robot unit; (B) – Formulation vessels; (C) – Rotor stator at the bottom of formulation vessels

Results

Thirty seven formulations were produced, including a number of repeats. The viscosity of samples was tested inline to ensure the ink was within printing range for an inkjet printer i.e. 4 – 12 mPa.s. Drying times of the inks were tested using auto-thermogravimetric analysis (TGA); volatile formulations are unsuitable for inkjet printing so higher drying times were preferred.

Stability against sedimentation was measured using light transmission at 650 nm; higher light transmission at the top of the vial was related to particles settling out.

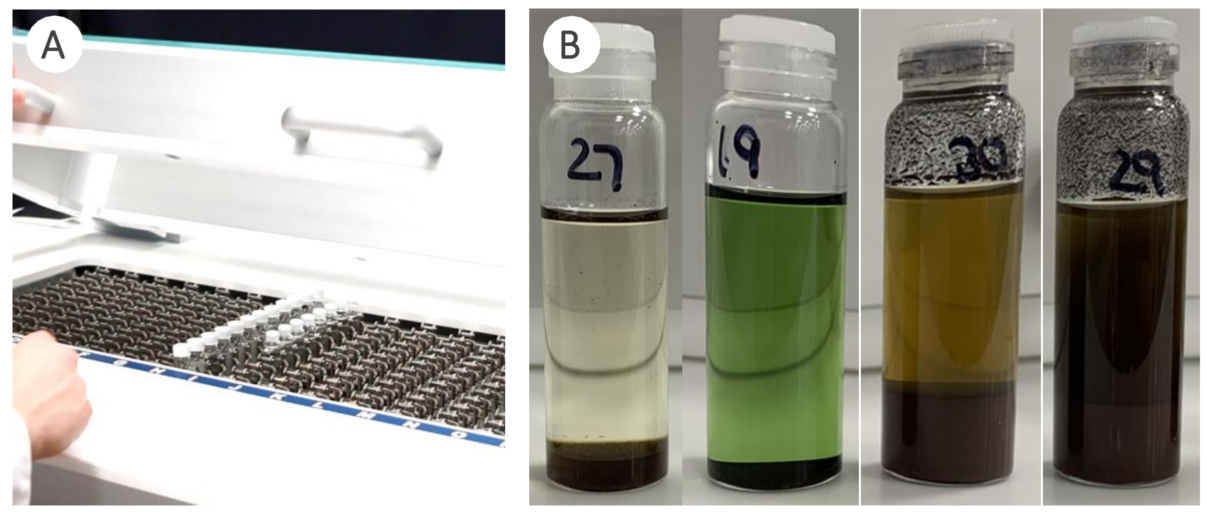

Figure 4 (A) – Robotic system used to test formulation stability; (B) – Examples of formulations after stability testing ranging from poor stability (left) to good stability (right)

Inks were dropped onto a microscope slide, dried and imaged using an optical microscope in reflection/DIC mode. Sporadic drying patterns (B) were associated with Marangoni flows, which we were aiming to minimise. Some inks formed a homogenous droplet (A) suggesting those solvent mixtures resulted in optimum drying.

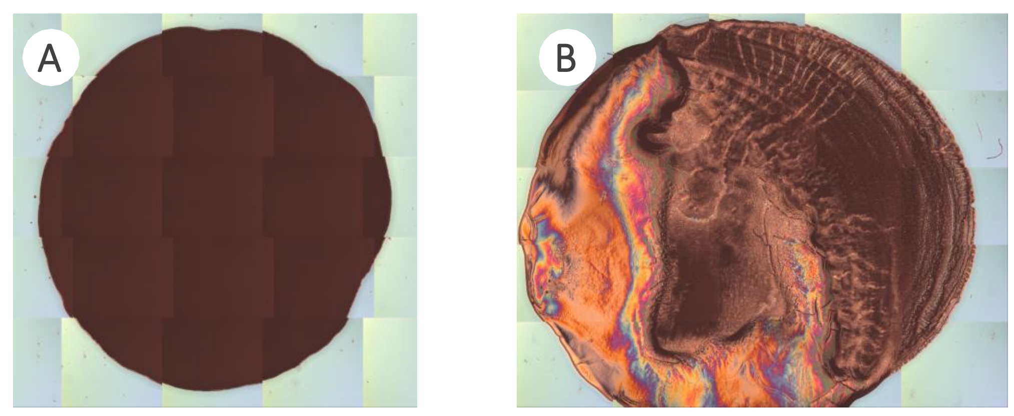

Figure 5 (A) – Stitched microscopy image of drop with good drying properties; (B) – Stitched microscopy image of drop with poor drying properties

DOE Model

Data was input into JMP statistical software and a reliable model was achieved (R2 fit > 0.64). The suggested formulation was produced; test results were similar to values suggested by the model.

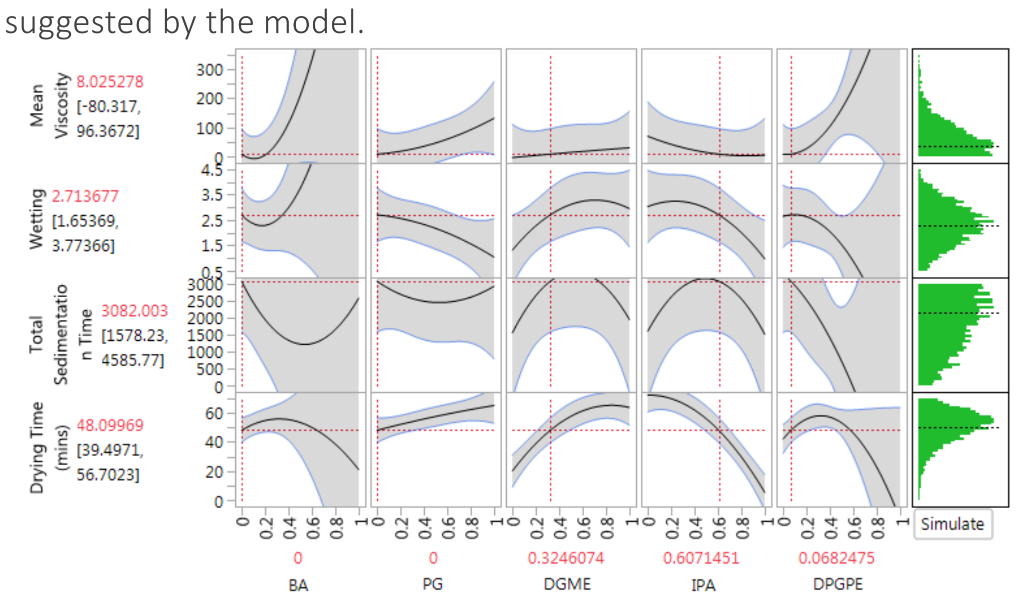

Figure 6 – Results of DOE; optimal formulation suggested to only contained DGME, IPA and DPGPE.

A

Mean Viscosity	15.8
Wetting	2.0
Total Sedimentation Time	563
Drying Time (mins)	39.7

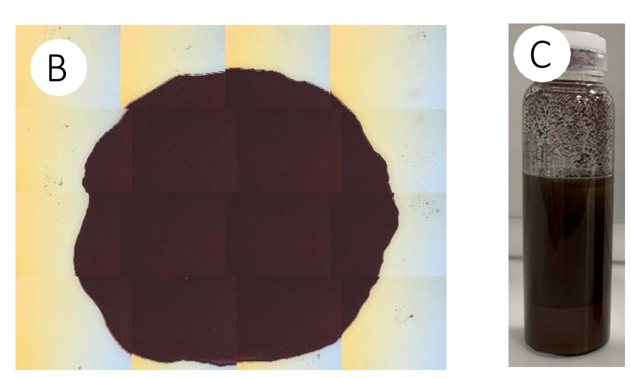

Figure 7 (A) – Test results for final formulation; (B) – Microscopy image of final formulation; (C) – Final formulation after stability testing

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