Data Driven Development of Inkjet Formulations using Design of Experiments

Veerle Goossens

CHEMSTREAM
SUSTAINABLE CHEMISTRY

Chemstream: The Chemical R&D Company

Mission

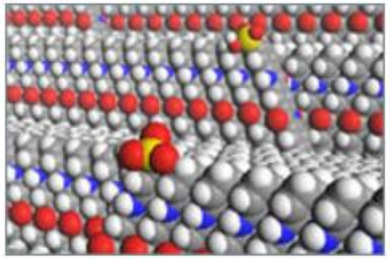
To translate customer requirements into chemical formulations with dedicated functionalities, from **design to prototyping and implementation**

Chemistry

- Crystal, Colorant and Dispersant Design
- Organic Synthesis
- Photo chemistry
- Colloids and Particle Surface Chemistry
- Interfacial chemistry, Wetting & Adhesion

Technology

- Dispersion technology
- Coating, Printing, Jetting
- Radiation curing (UV, UV-LED, e-Beam)
- Atmospheric Plasma



Methodology

- Molecular modeling
- Experimental design (DOE)
- High throughput screening

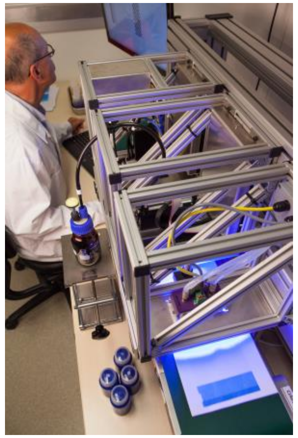
Analytical and physical chemical tools

- UV-vis, FTIR, GCMS
- Particle size distribution
- Contact angle, Surface tension, Viscosity

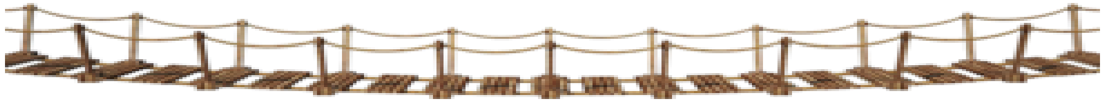
CHEMSTREAM
SUSTAINABLE CHEMISTRY

Innovation is a Dynamic Collaboration between R&D and Technology

Ink design
- Mechanical properties
- Process characteristics
- Colour (pigment / dye)
- Hydrophobicity
- Rheology
- Legislation
- …



Technology
- Type of printhead
- Resolution printhead
- Printing speed
- Belt speed
- Curing method (LED/UV, Thermal,…)
- …



CHEMSTREAM
SUSTAINABLE CHEMISTRY

Bridging the gap between Chemistry and Technology
- Ink / Media interaction
- Ink / Printhead interaction
- Process parameters
- Substrate pretreatment
- ….

Data Driven Approach

Identify
- Understand customers' needs and wants
- Translate them into technical requirements

Set- up
- Repeatable & Stable
- Easily quantifiable
- Select factors and ranges

Collect data
- Perform experiments
- Screening experiments to discover most influencial factors
- Optimization experiments to optimize factor levels

Analyse
- Visual assesment of the data
- Fit model to data
- Interpret results
- Use model to predict outcome

Test
- Prototypes testing in real life conditions

Design of Experiments

Design of Experiments

One Factor At a Time (OFAT)

- Very **intuitive**
- Test 1 possible cause if it doesn't work go to the next one
- Compare **individual values**
- Optimize the system by optimizing one factor at a time

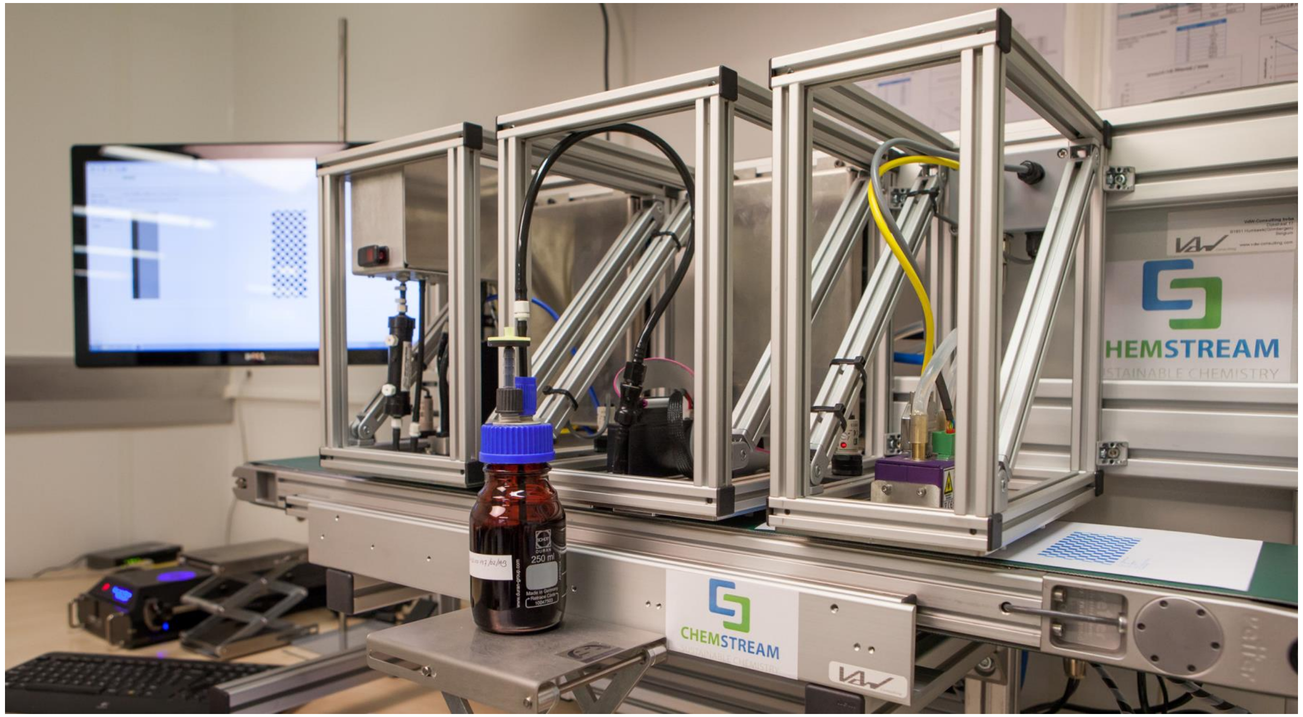
Design of Experiments (DOE)

- All parameters are changed together **simultaneously**
- Comparison of **averages**
  - **Greater accuracy** in effect estimates
  - Influentual factors **more likely to emerge** from environmental noise
- Estimation of **interactions**
- **Balanced design**
  - Causation vs Correlation

Technology

- Type of printhead
- Resolution printhead
- Printing speed
- Belt speed
- Curing method (LED/UV, Thermal,…)
- Type of substrate
- Process parameters
- ….

MPU
Modular Printing Unit

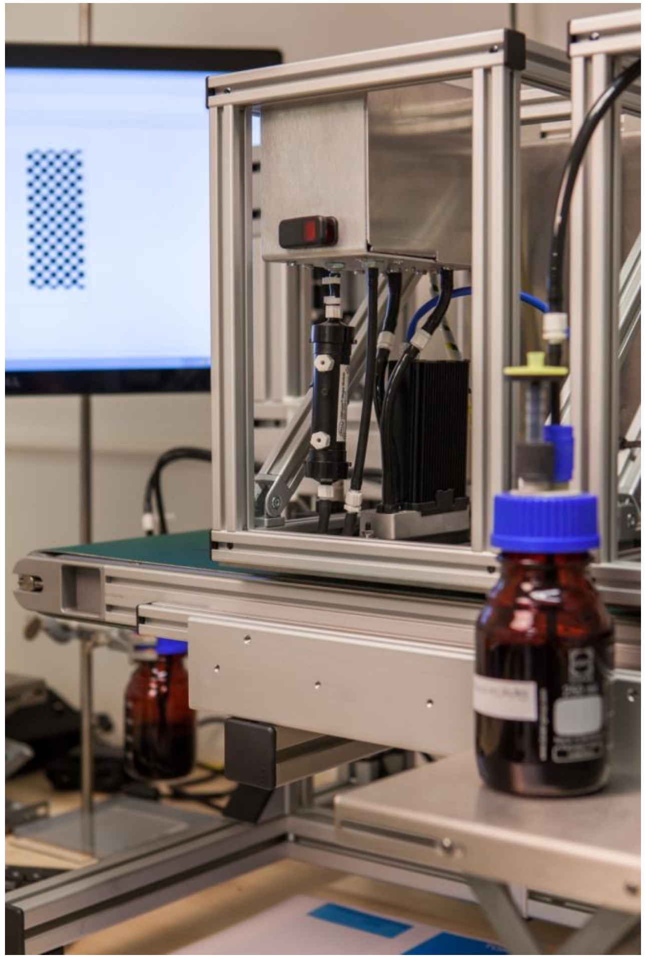


in cooperation with

VAV Consulting

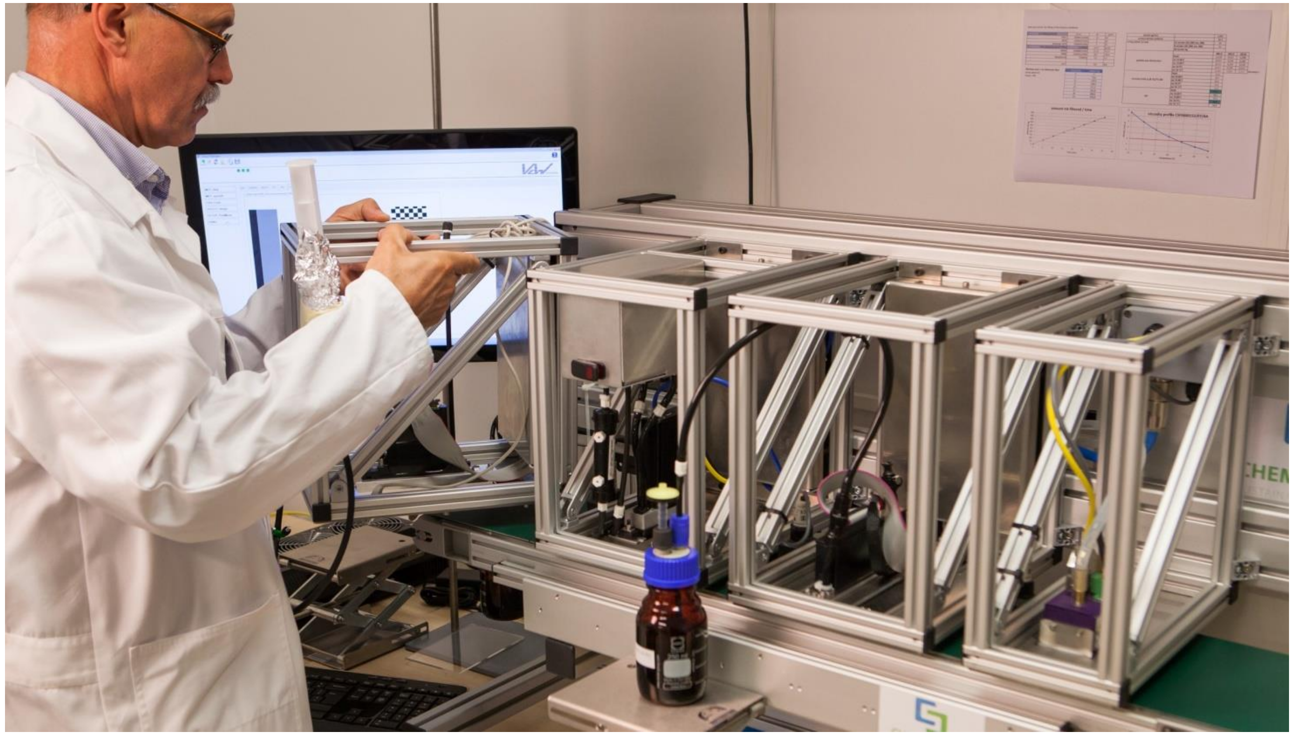
Modular printing unit: easy tool for feasibility research

- Mimic of an in line coating/printing process
- Fast iterations of ink prototypes
- Fast iterations with different printheads
- Low investment level for customer
- Ink/media interactions
- Easy to provide test samples



MPU:

Easy removal and/or addition of printheads with their appropriate driver electronics

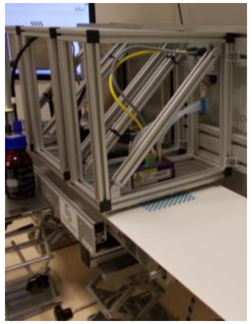


MPU

A variety of substrates:

- Plastics
- Metals
- Textiles
- Glass
- DCP
- 3D Objects
- …

PVC



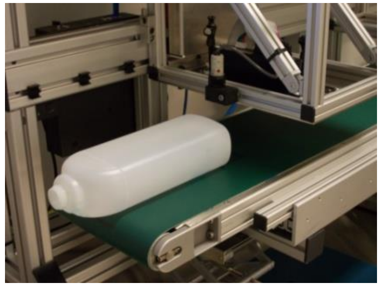
Cu-Tube

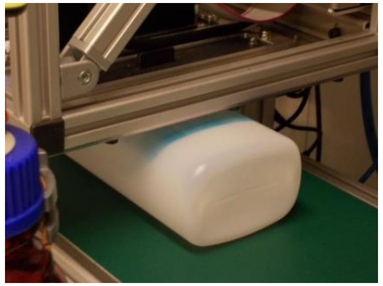
Al-profile

Cotton

SiN/Glass

DCP:
Direct to Container Printing

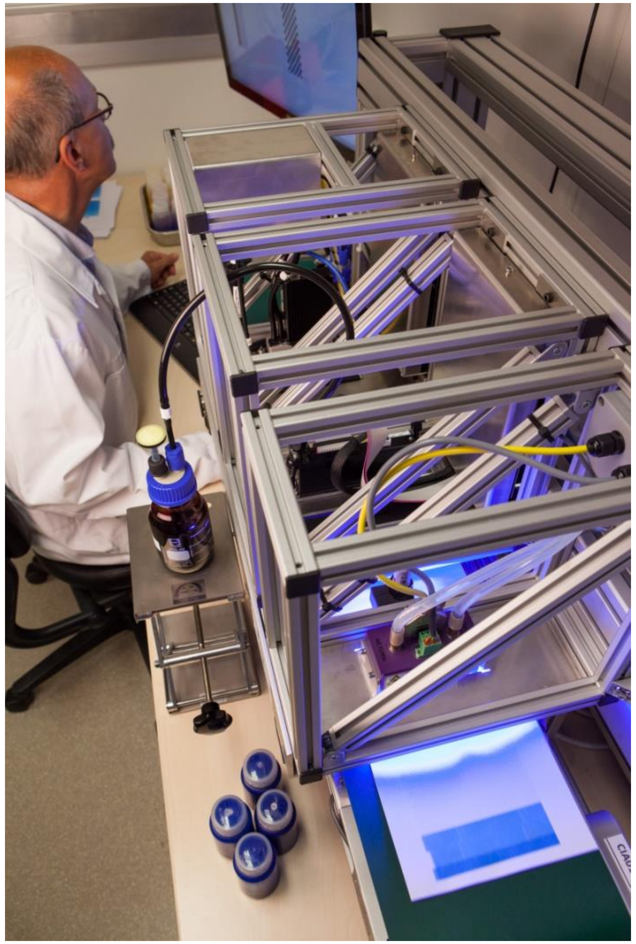




MPU: Features

- ‘Down to earth’ printing system
  - Table top dimensions
  - No special infrastructure
  - Belt based system for single pass
  - Potential for crude multi pass
  - Small ink amounts

- Flexible towards:
  - Head selection and replacement
  - Substrate height
  - Print strategies
  - Ink replacement



Ink design

Responses

- Mechanical properties
- Process characteristics
- Colour (pigment / dye)
- Hydrophobicity
- Conductivity
- Rheology
- Legislation
- Ink / media interaction
- Ink / printhead interaction
- …

Factors

- Monomers
- Photo-initiators
- Inhibitors
- Pigments dispersions
- Functionalizing agents
  - Hydrophobing
  - Conducting
  - …
- …

Ink design

Screening designs

- To examine many factors at a time and identify those that have the greatest effect on the response
- Identify the design space
- Only main effects are studied
- No interactions

Optimization designs

- Limited amount of factors
- Interpolation in the design space
- Locate the sweet point: Optimum for all responses
- Investigate interactions between factors

Example – LED curable ink: initiation system

Step 1:

- Selection of initiators based on the correlation between their absorption bands and the curing system (bulb / LED)
- Price, availability, health/safety,…

Step 2:

- Screening experiment (in/out) in matrix
  - Cure speed, yellowing, compatibility with matrix,…

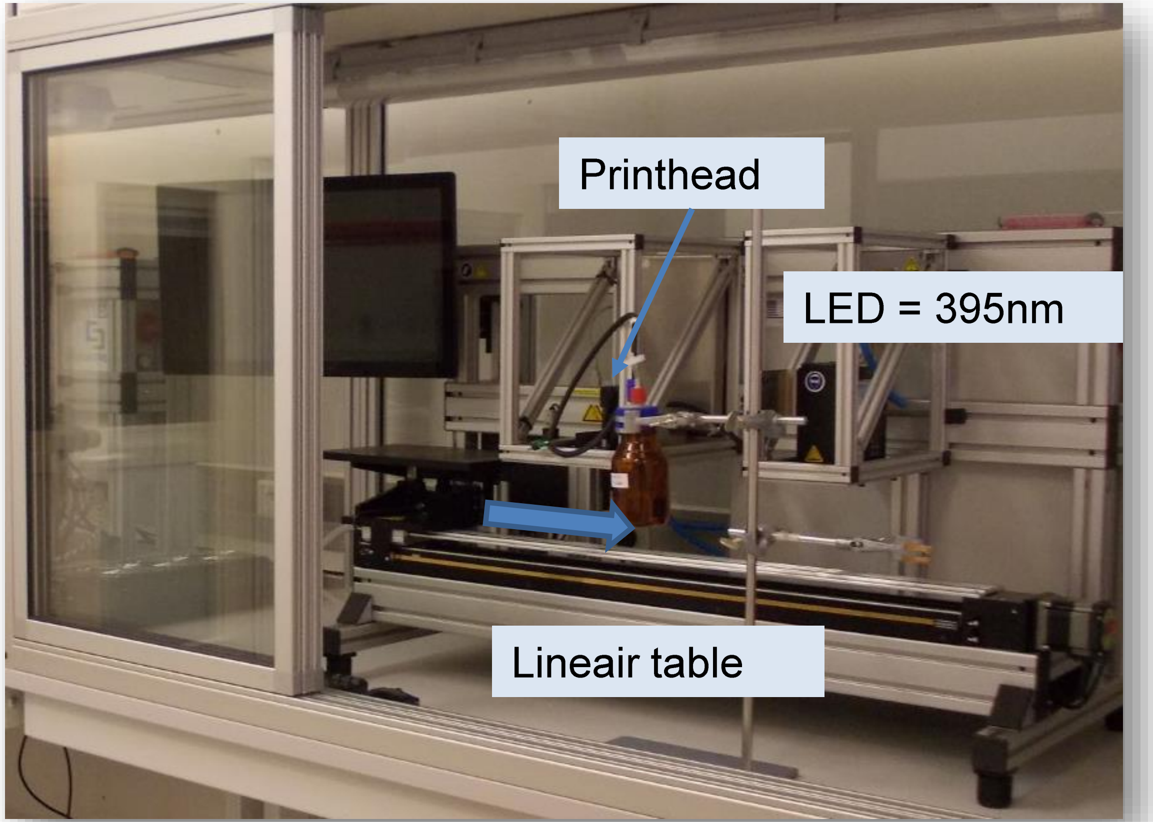
Step 3:

- Optimization of initiator concentration
  - Selection of limited # initiators
  - @ different concentration levels
  - Interactions between initiators?

Example – LED curable ink: initiation system

Experiment:

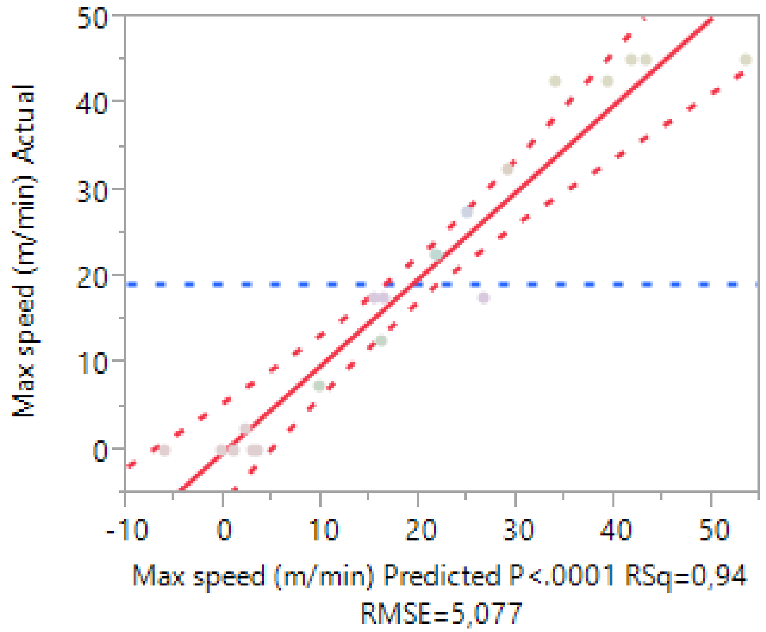
- Initiation system: TPO – DETX
- 12μm coatings on glass slides
- 395nm LED
- Advancing speeds
- Swab test to confirm polymerization



Example – LED curable ink: initiation system

TPO (%)	DETX (%)	Max curing speed (m/min)
10	5	45
7,5	0	12,5
7,5	2,5	15
10	2,5	42,5
2,5	10	15
2,5	7,5	15
5	10	32,5
7,5	10	45
7,5	7,5	42,5
0	5	0
2,5	0	2,5
2,5	2,5	7,5
5	5	28,5
10	0	22,5
10	10	45
0	10	0
0	2,5	0

Actual by Predicted Plot



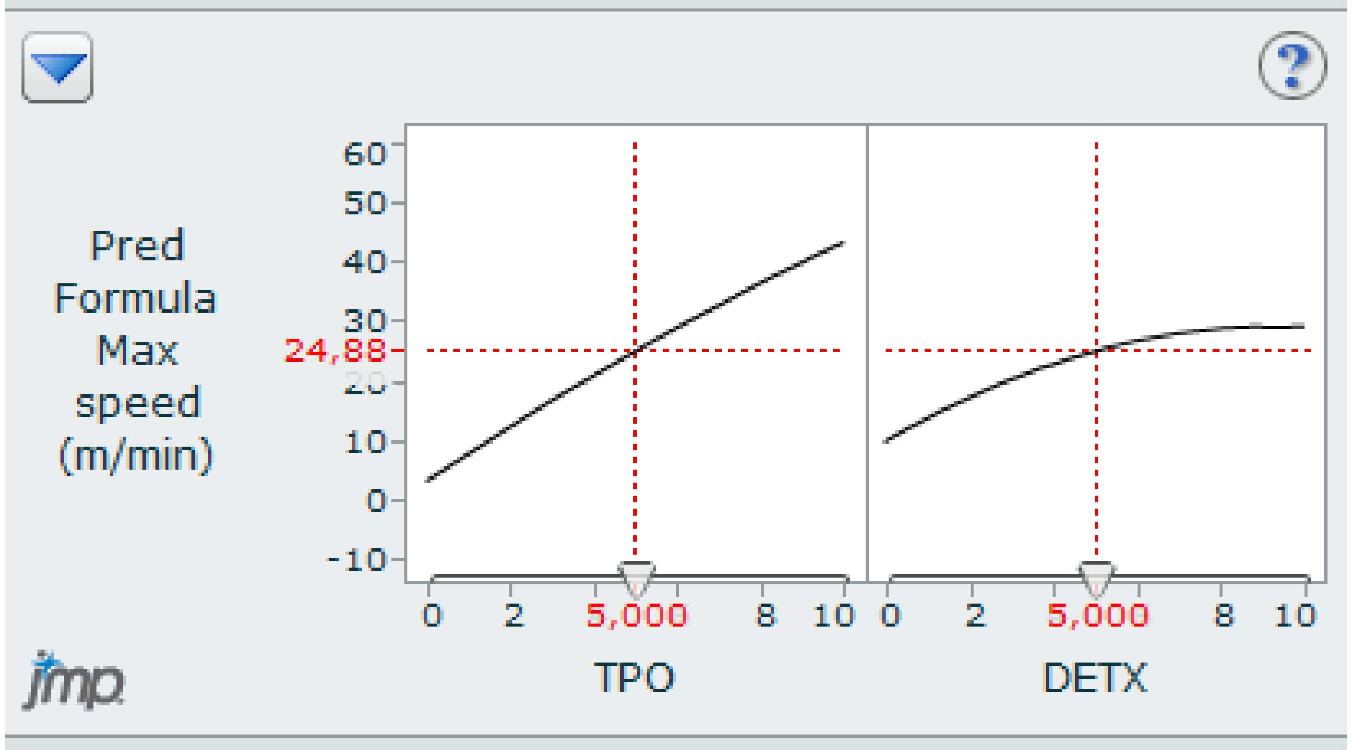
Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	5	5427,8901	1085,58	42,1163
Error	14	360,8599	25,78	Prob > F
C. Total	19	5788,7500		<,0001*

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	24,875131	2,607429	9,54	<,0001*
TPO(0,10)	20,082242	1,545903	12,99	<,0001*
DETX(0,10)	9,6765376	1,486081	6,51	<,0001*
TPO*DETX	6,1789522	2,084945	2,96	0,0103*
TPO*TPO	-1,763151	2,769165	-0,64	0,5346
DETX*DETX	-5,540252	2,72972	-2,03	0,0619

Example – LED curable ink: initiation system

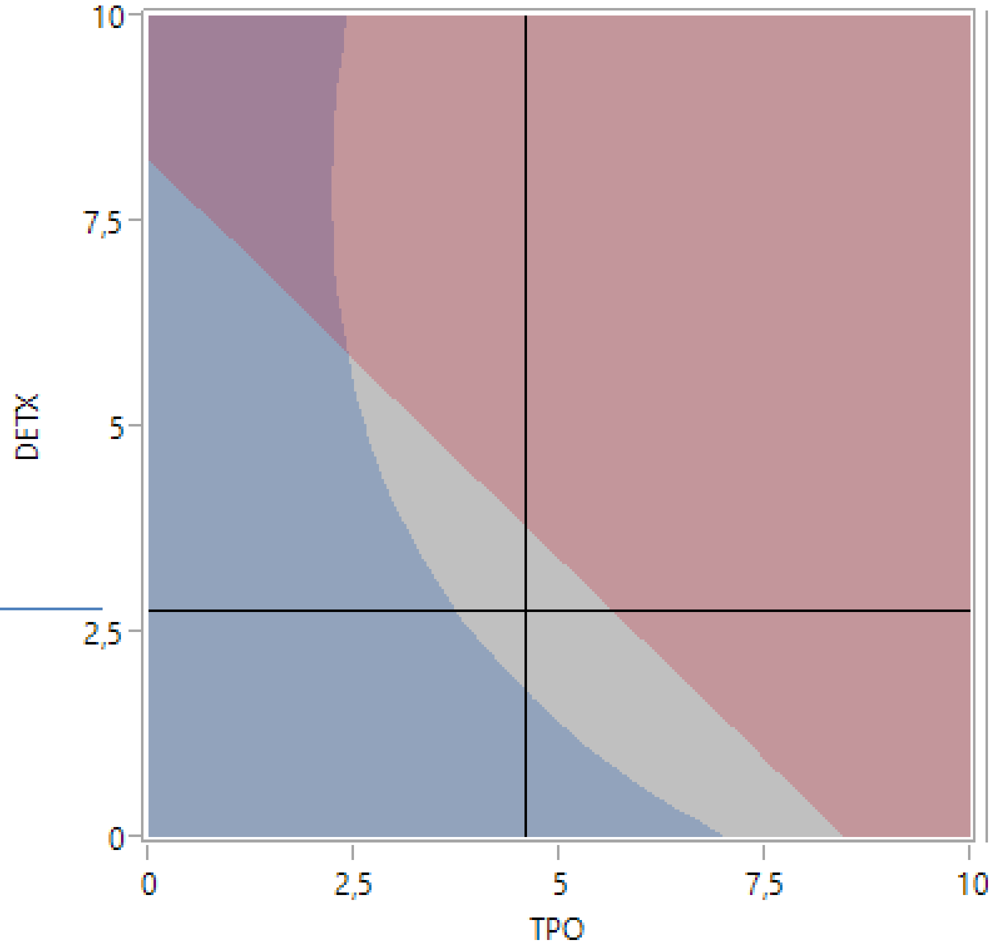


Example – LED curable ink: initiation system

Optimizing 2 responses simultanously

- Cure speed higher than 15m/min
- Price /kg below 3€

Price = 2,6€
Cure speed = 18m/min
TPO = 4,5%
DETX = 2,8%

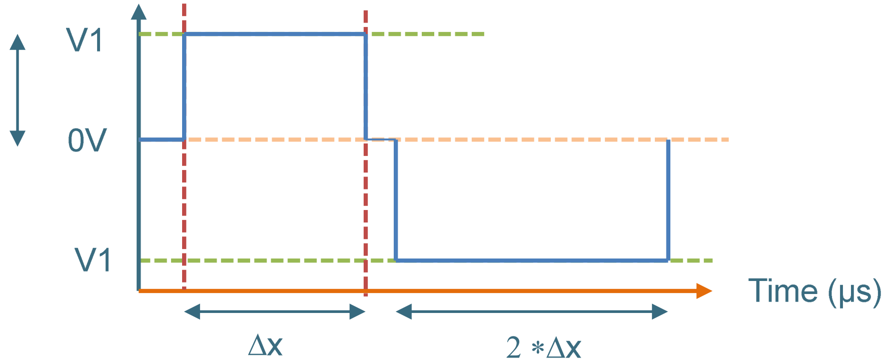


Drop Formation

- Avoid satellites
  - Prevent fluid build up on nozzle plate
  - No trailing
- Improve print quality
  - Control drop size and mass for optimal resolution
  - Constant drop speed for fire accuracy

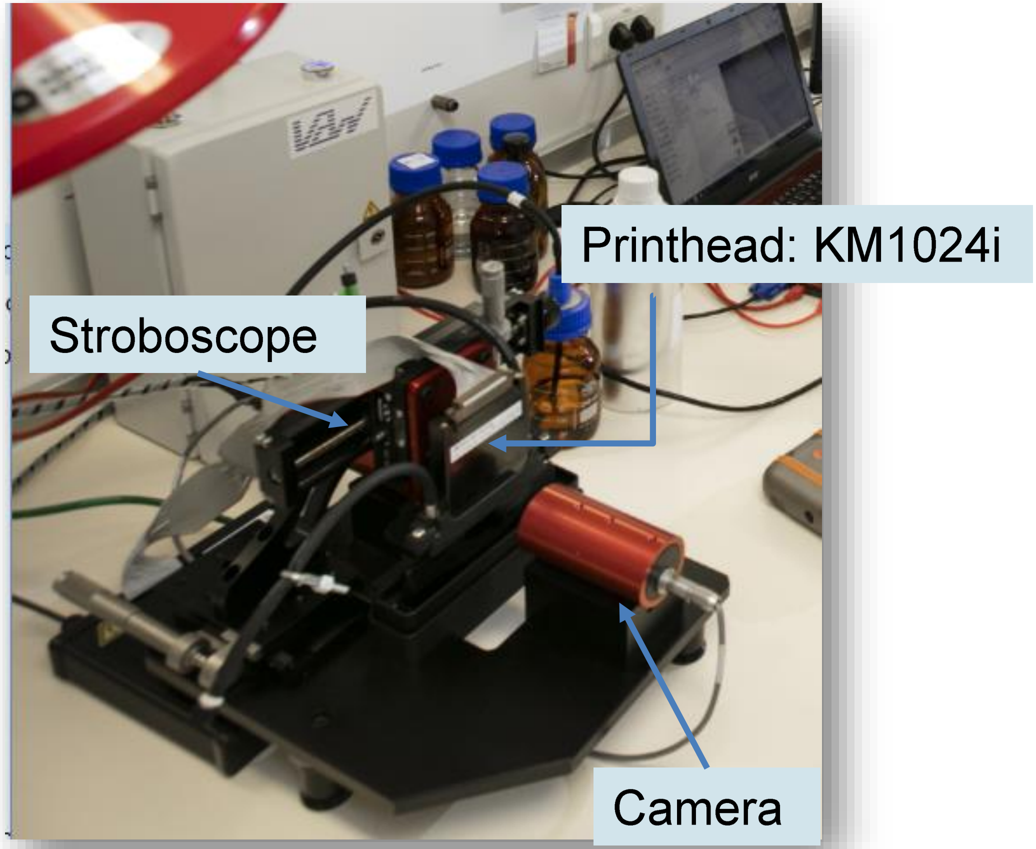
Drop Formation

- Experimental parameters
  - **Viscosity** of the ink (by controlling jetting T)
    - Influences the propagation of the pressure wave in the nozzle
  - **Waveform**
    - Voltage = Height of pulse = **V1**
    - Pulse Width= **Δx**



Drop Formation

Experimental set-up



Setting: 1dpd

Predictors:

Voltage: 9V – 11V -13V

Δx: 1,6µs ≤ Δx ≤ 5µs

Viscosity: 8,5cP – 10,5cP – 13cP

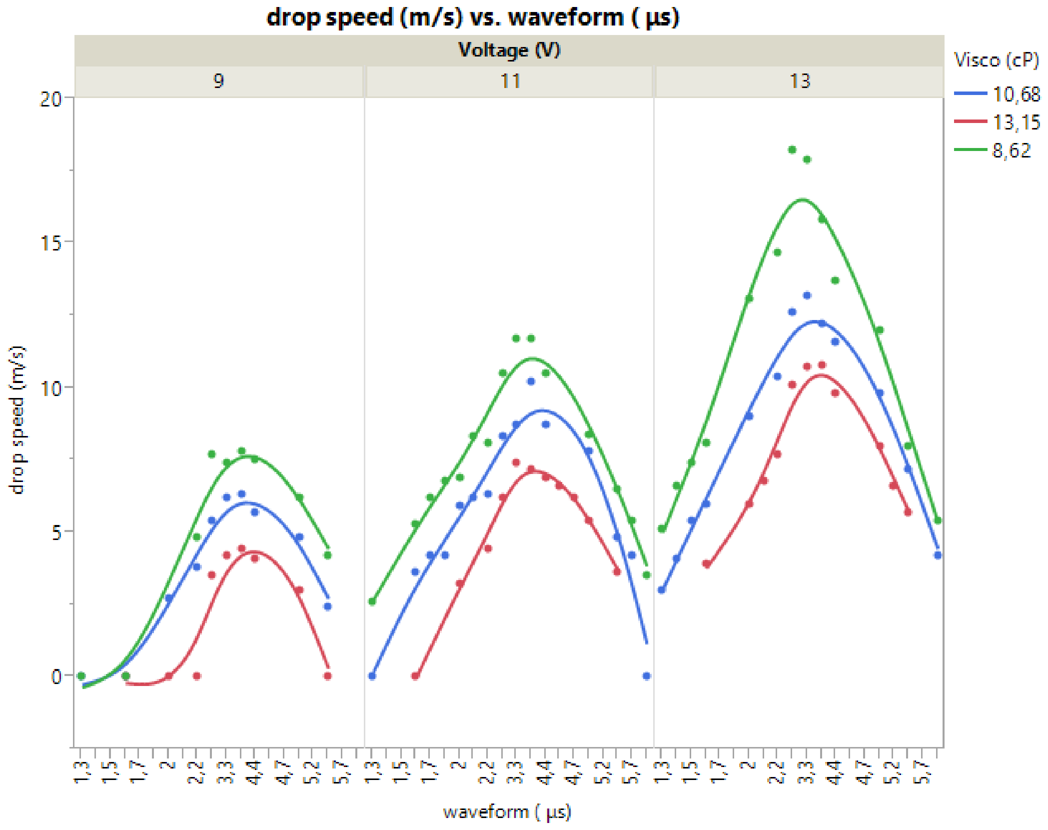
Responses:

Drop speed

Satellites

Tail length

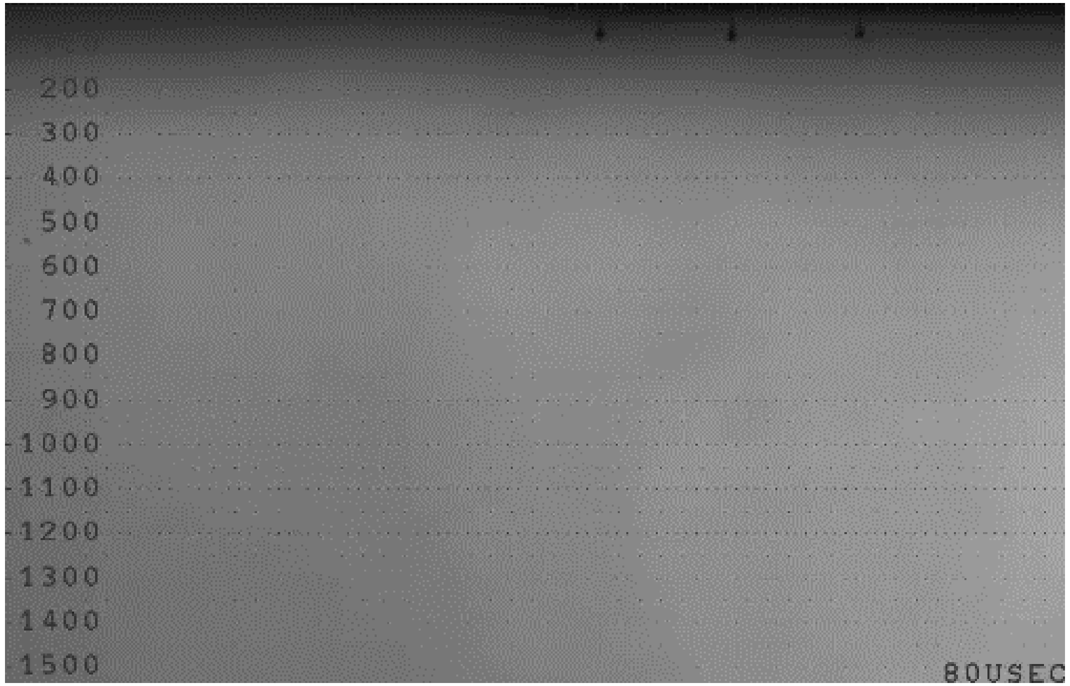
Drop Formation



Visual assessment of data

- Drop speed increases as a function of Voltage
- Drop speed increases as Viscosity decreases
- Parabolic relationship between drop speed and waveform. Maximum @3,8μs
- Similar observations for Tail length and #satellites

Drop Formation



Parameters:

- 9V
- 10,8cP
- 3,8μs pulse width

Output

- Drop speed = 6,3m/s
- Tail length = 147 μm
- No satellites

Conclusion

Ink development: a dynamic iteration process between 3 sub processes

- Chemical formulation process
- System integrated process
- Customer driven interaction process in an open environment

Efficient implementation of these 3 processes in a lab environmental workflow leads to:

- Short development times
- Low investment level (during feasibility phase)
- Dedicated high performance end user requirements

Thanks for listening

You are invited at our booth for further information and discussions:

- Els Mannekens
- Veerle Goossens
- Frank De Voeght

CHEMSTREAM

More info on our website:
www.chemstream.be