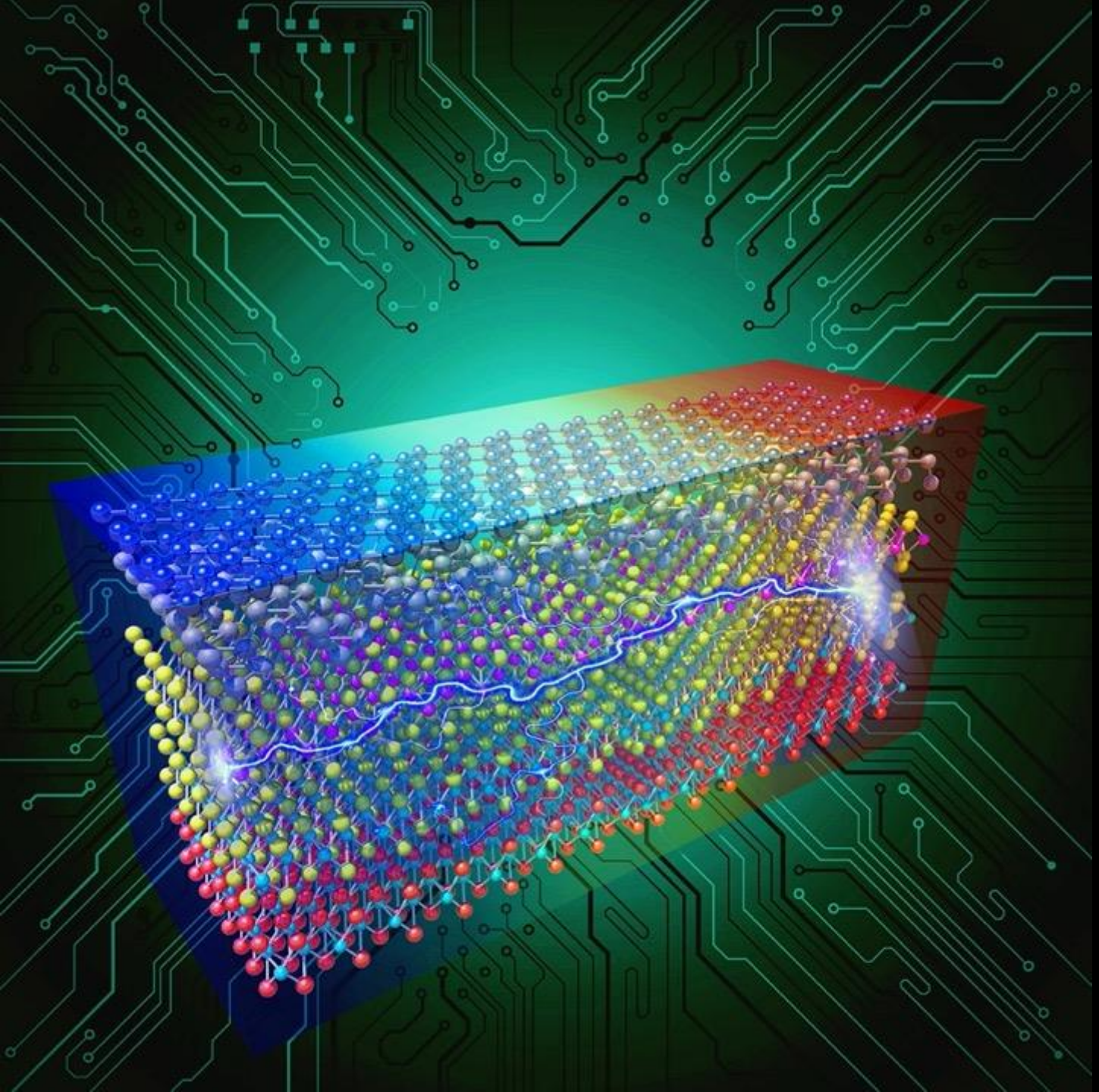

AI and High-Throughput Testing to Accelerate Catalyst Discovery

Kedar Hippalgaonkar

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Nanyang Technological University,
Agency for Science Technology and Research
Xinterra

27 June 2024

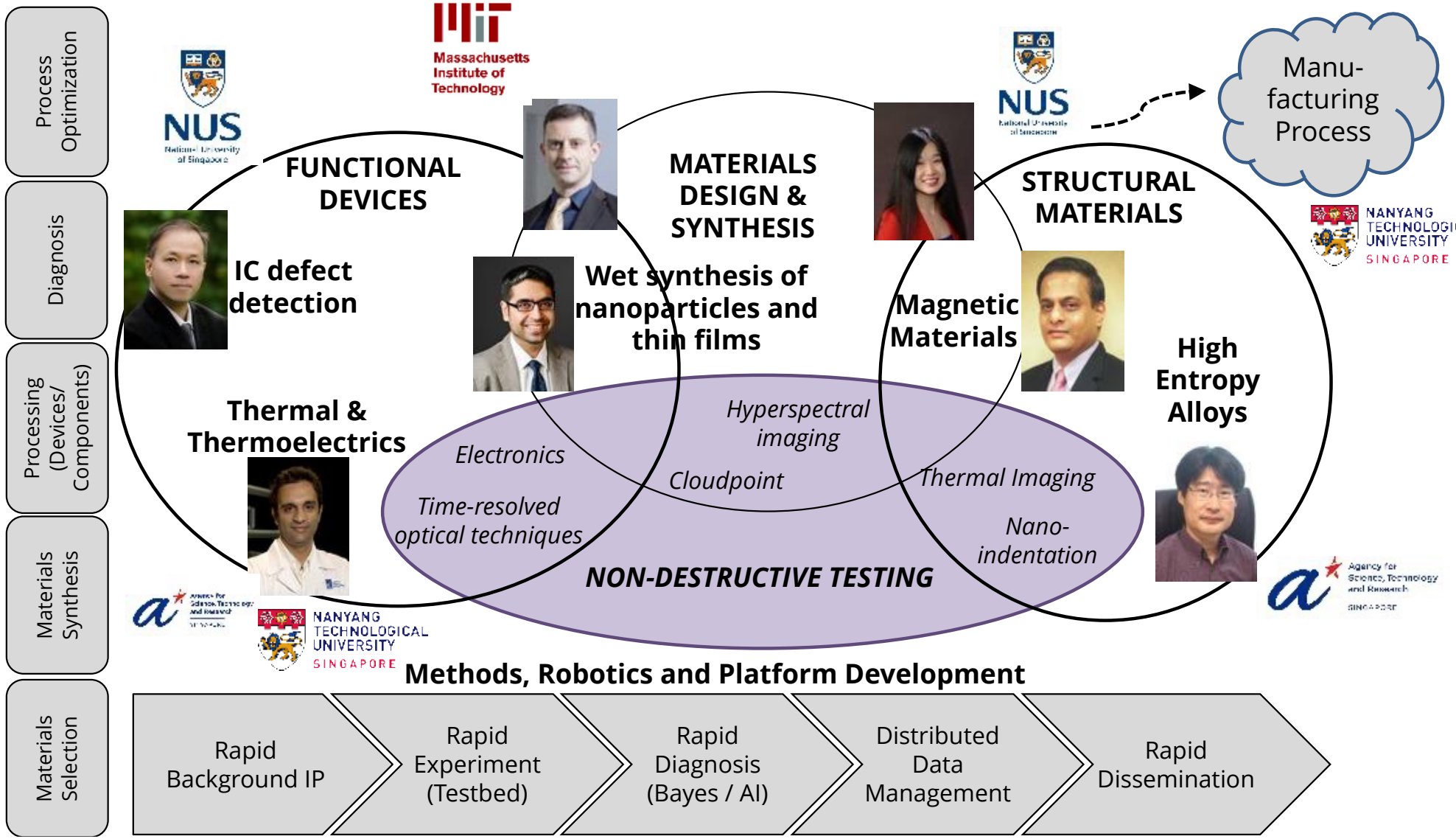
NANYANG TECHNOLOGICAL UNIVERSITY SINGAPORE

Agency for Science, Technology and Research SINGAPORE

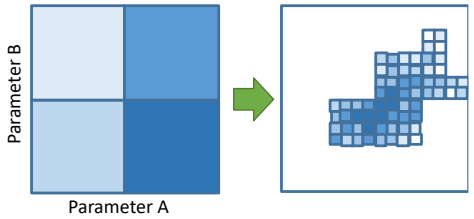
ACCELERATED MATERIALS DEVELOPMENT FOR MANUFACTURING

SINGAPORE

US$18M 5-year Research Program on Accelerated Materials Development

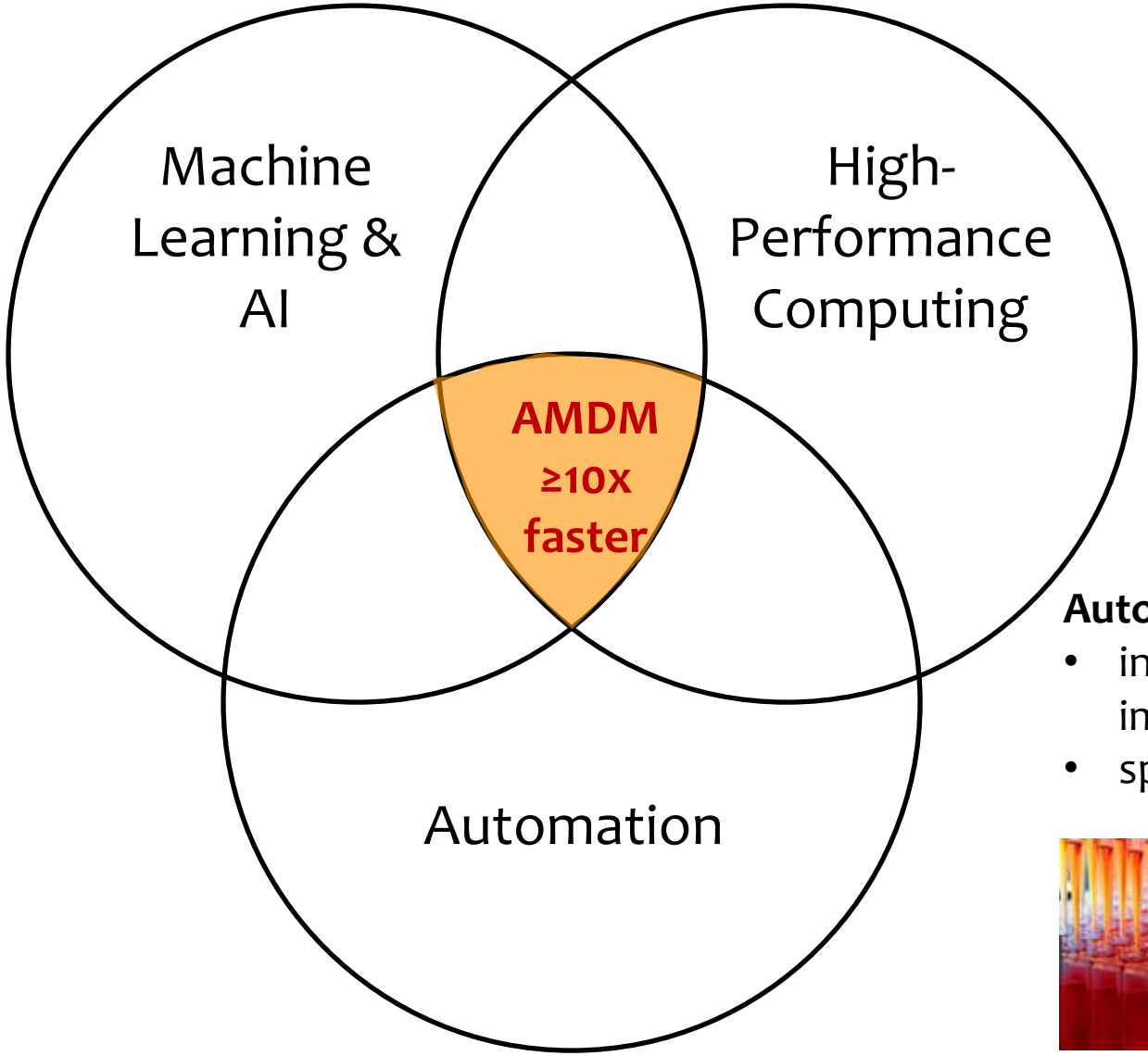


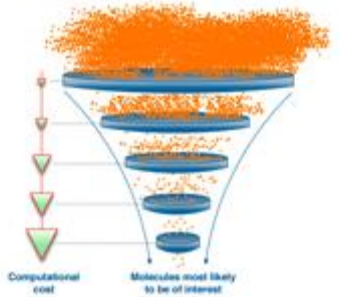
Convergence Enables Accelerated Materials



ML/AI:

- converts experimental data into actionable intelligence
- quickly navigates complex manifolds
- cognitive assistant



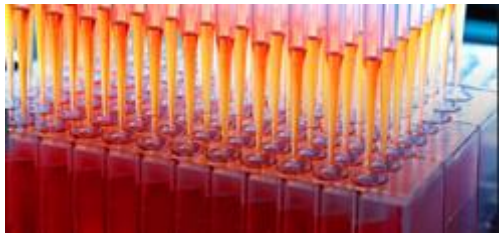


HPC simulation/screening:

- Materials Data
- focuses limited experimental bandwidth on most promising candidates

Automation/robotics:

- increases reproducibility, improving signal-to-noise
- speeds up experimentation

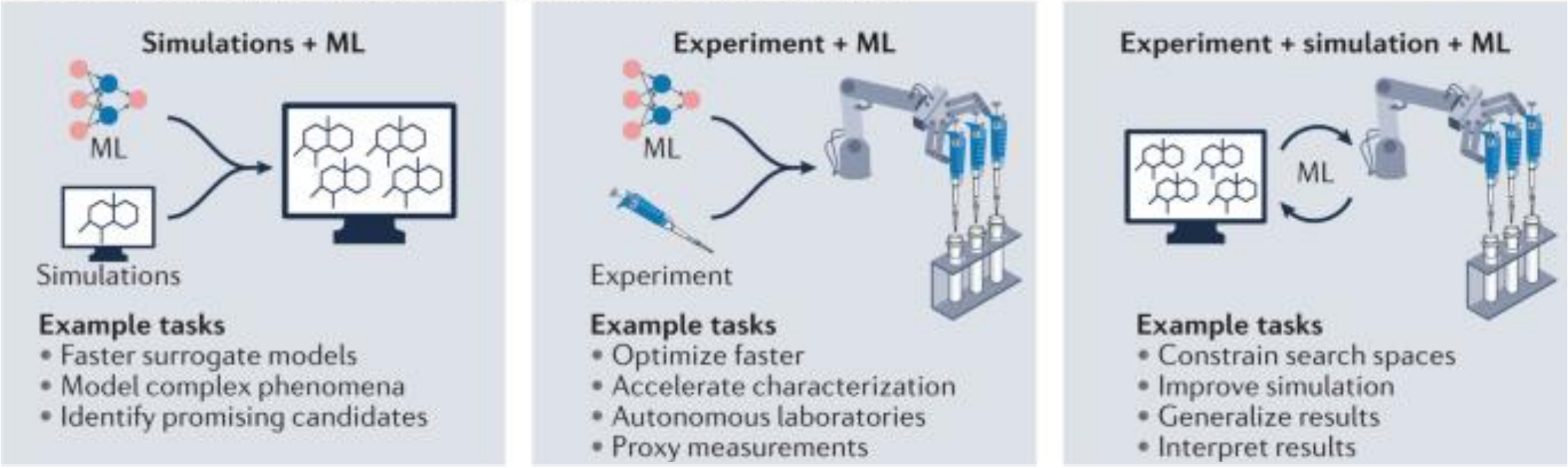


S.V. Kalinin *et al.*, *NMat* **14**, 973 (2015)
Y. Liu, *J. Materiomics* **3**, 159 (2017)
D.P. Tabor *et al.*, *Nature Reviews Materials*

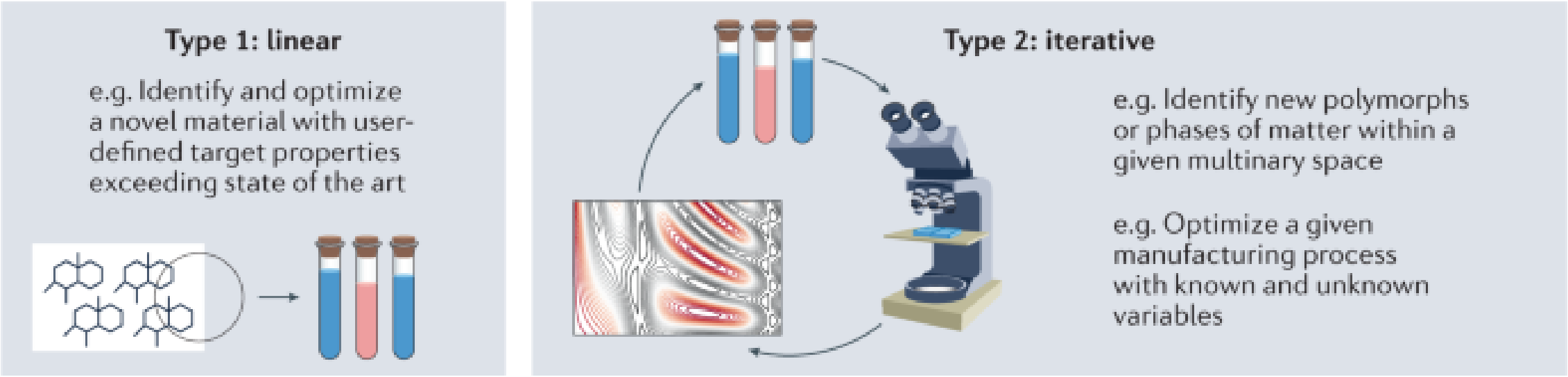
https://arxiv.org/abs/1803.11246
https://www.cifar.ca/wp-content/uploads/MissionInnovationIC6Report.pdf

CREATING GROWTH, ENHANCING LIVES

a Toolset integration: integrate ML with (autonomous) research tools

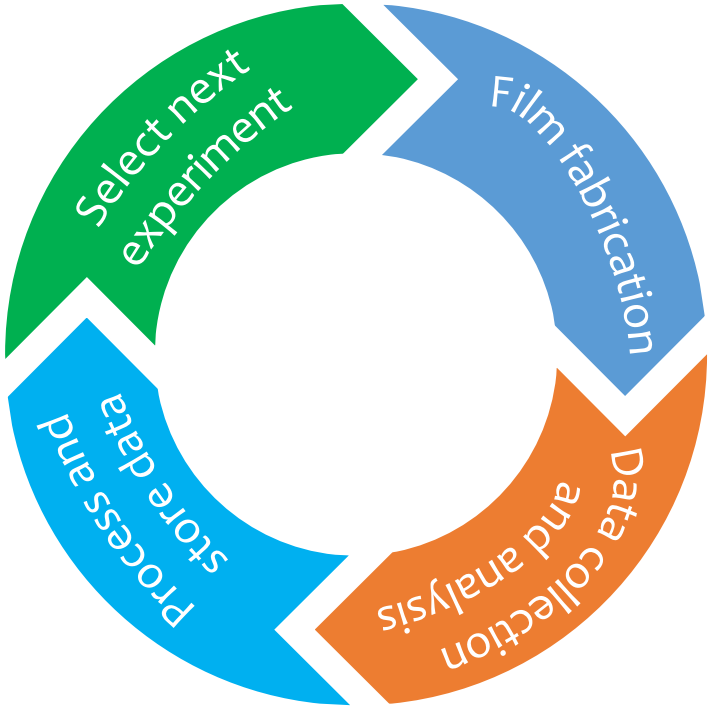


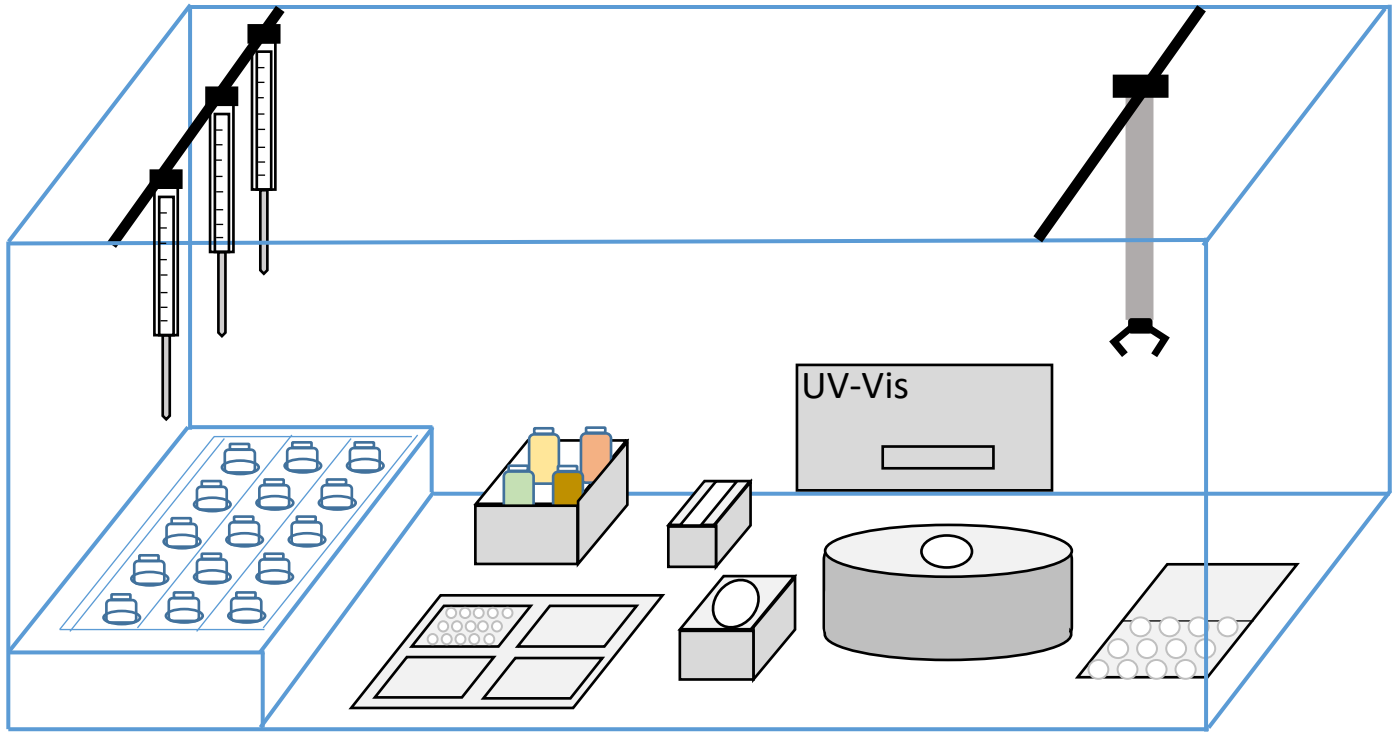
b Workflow integration: integrate discrete ML-enabled (autonomous) research tasks



K. Hippalgaonkar, Q. Li, T. Buonassisi et. al. **Nature Reviews Materials (2023)**

Example #1: Optimization of Cu-Sb-S for photoelectrochemistry

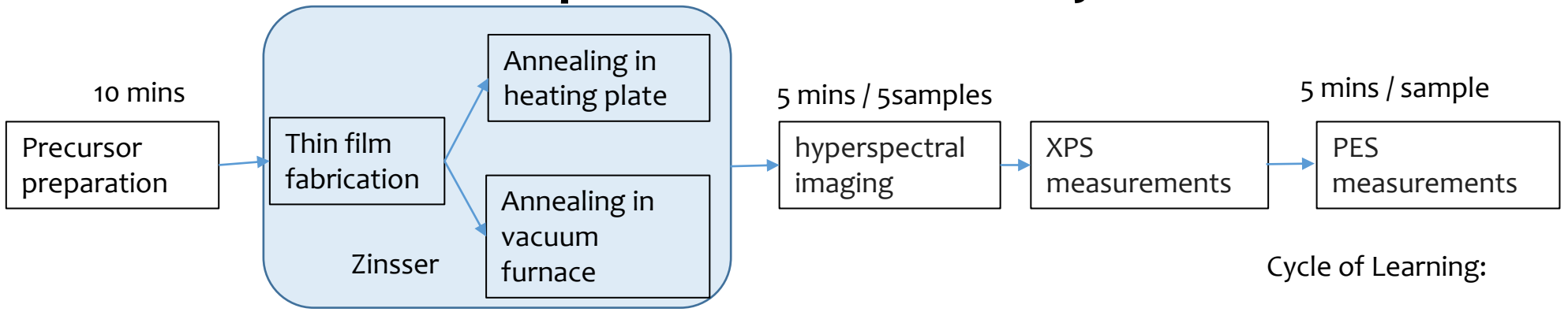




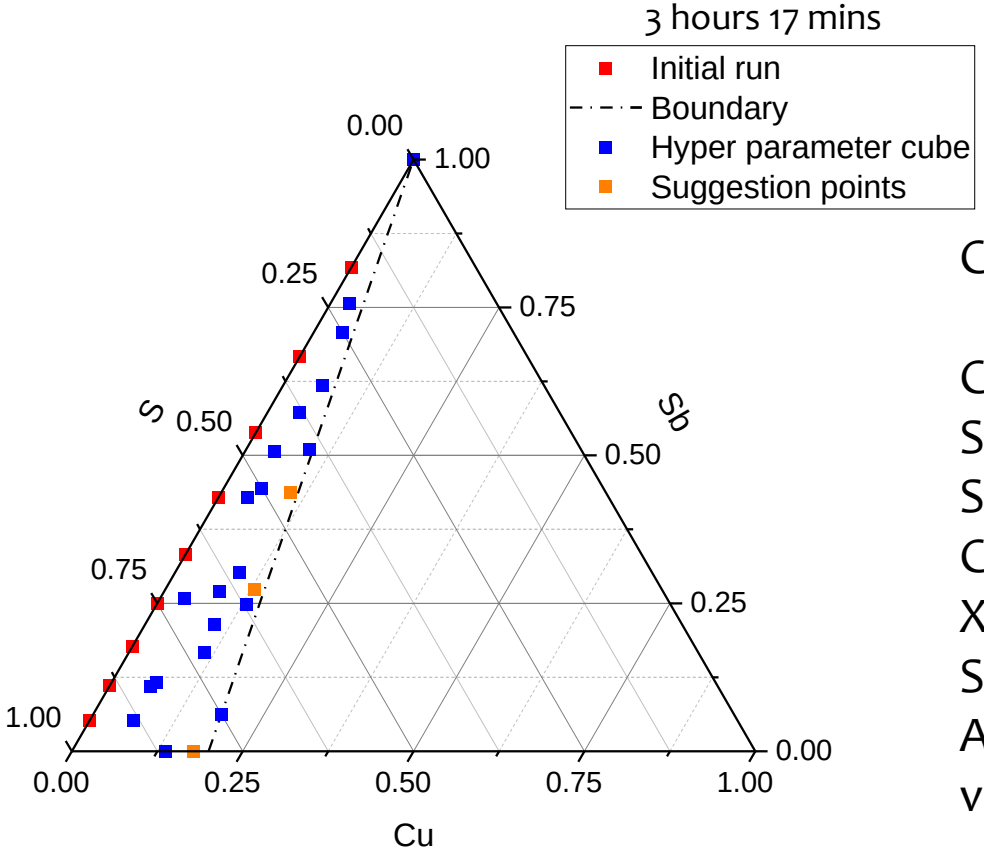
High-fidelity optimization for the best sample from ML-guided materials screening

Bai Yang et. al. ***Advanced Materials*** 36, 2 , 2304269 (2023)

Optimization of Cu-Sb-S for photoelectrochemistry



3 hours 17 mins



30 mins for each sample, 15 samples on one batch
1h preparation
So 5 samples will take 2h 50 mins

Cycle of Learning:

6 hours 30 mins for one batch (5 different samples, 3 repeats for each, total 15 samples, annealing)

Constraints

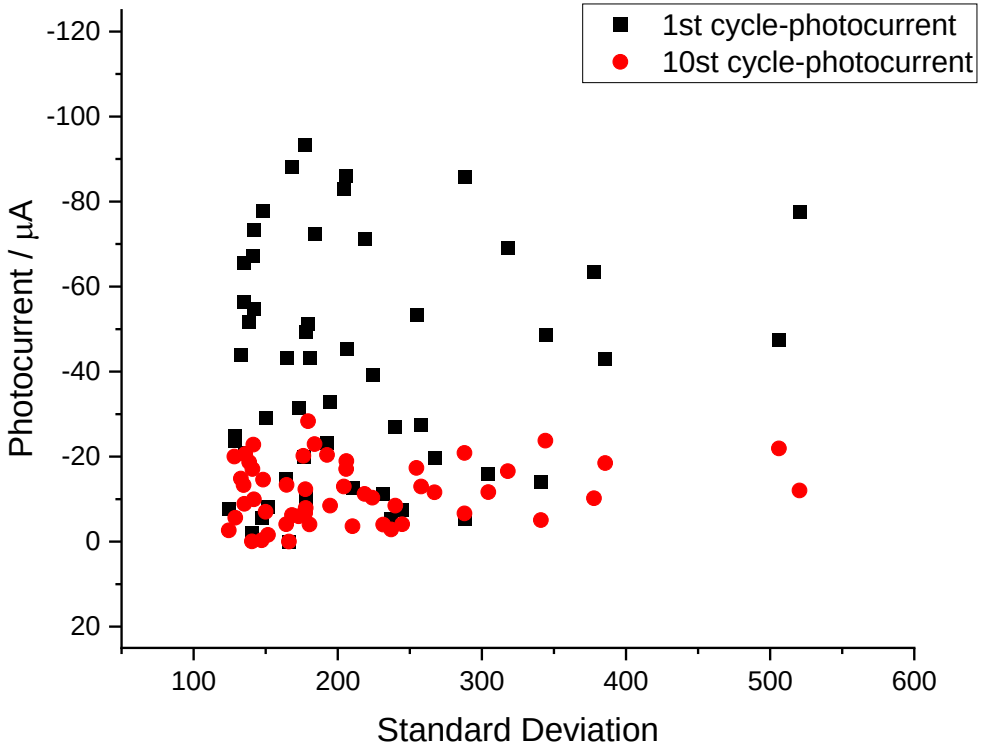
Cu: 0≤ X ≤ 2000
Sb: 0≤ Y ≤ 2000
S: 0≤ Z ≤ 2000
Cu ≤ S/4
X+Y+Z=2000
Spin speed: 2500-4000
Annealing: 150 °C in air or 200 °C in vacuum furnace

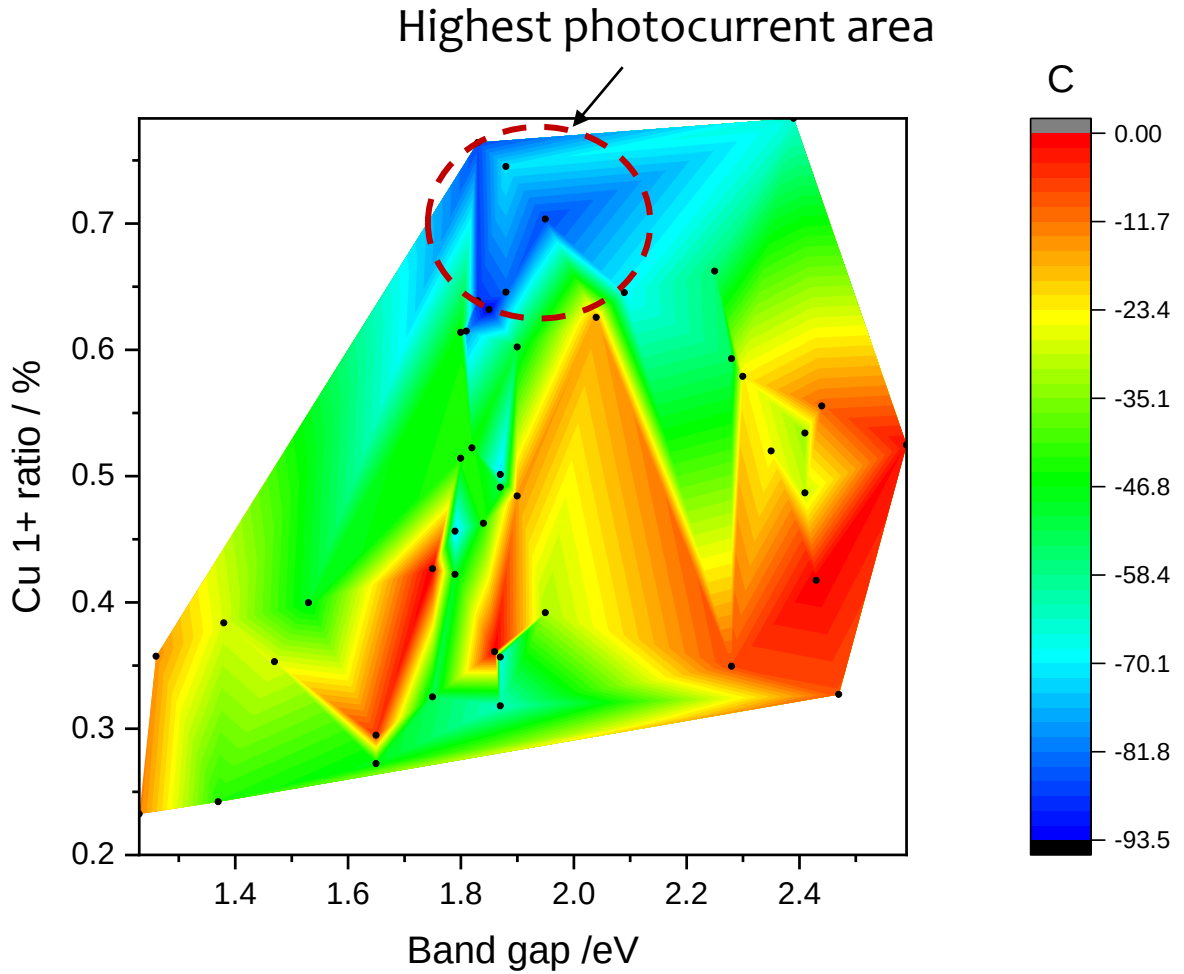
Multi-objectives

- **Optical gap ~ 2eV**
- **Highest Film Uniformity**
- **More Cu^{1+} percentage**

Bai Yang et. al. ***Advanced Materials*** *36, 2 , 2304269* (2023)

PEC Materials Discovery

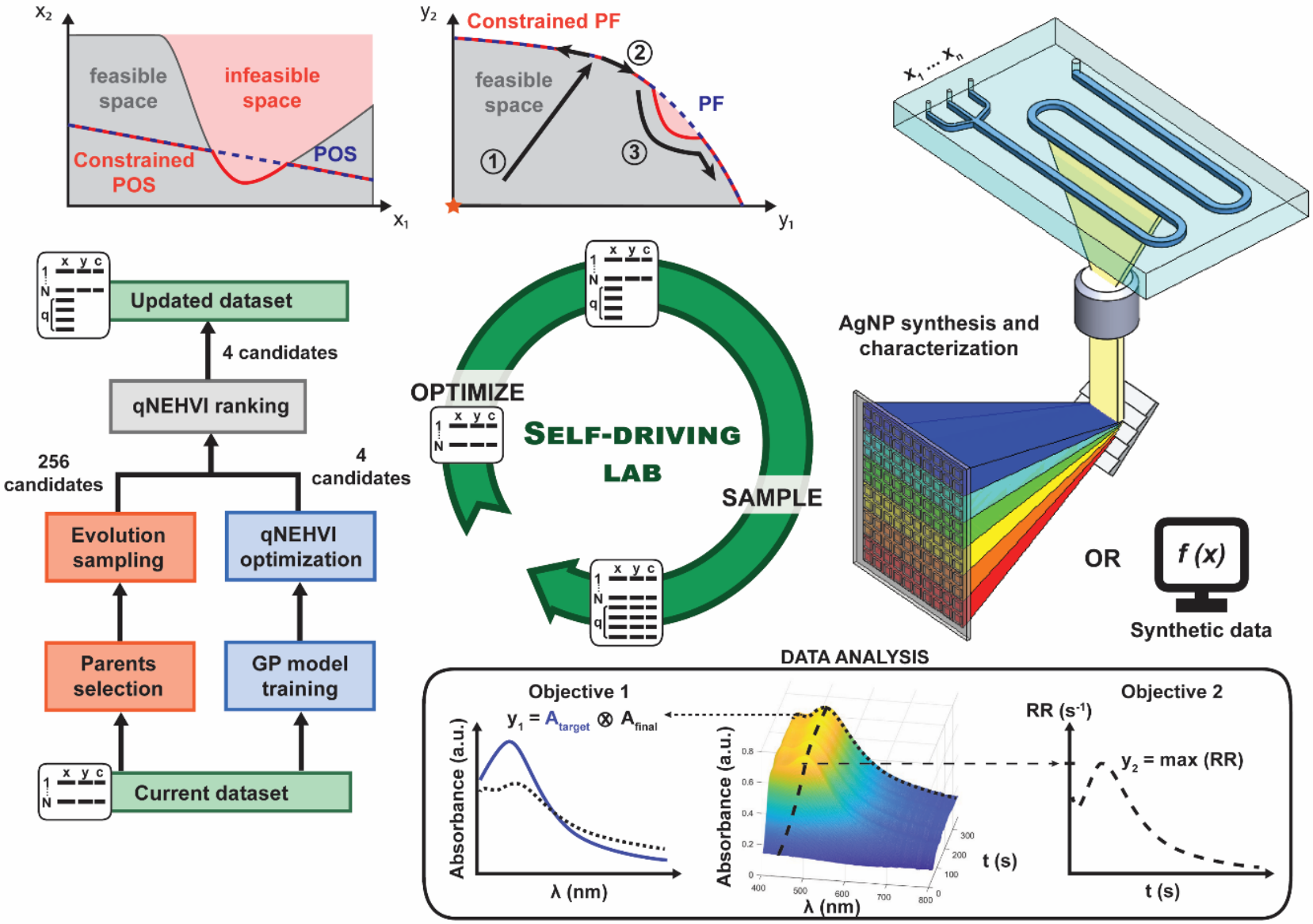




No clear corelation between the uniformity of sample films and photocurrent

Bai Yang et. al. ***Advanced Materials*** *36, 2 , 2304269* (2023)

Example #2:
NEW ALGORITHMS – Evolution-Guided Bayesian Optimization Self-Driving Lab

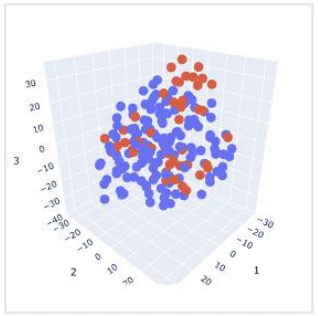


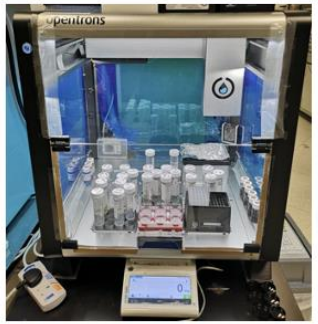
Andre Low, Flore Mekki-Berrada, Saif A Khan et. al. npj Computational Materials (2024)

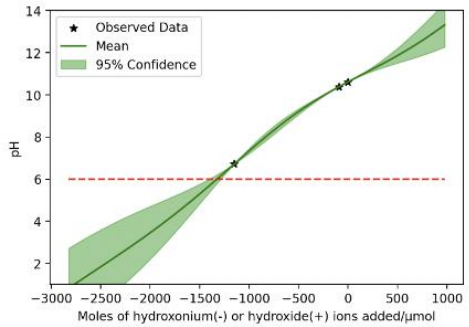
Example #3: Shampoo Formulations

ML-driven Design of Experiments

Intelligent DoE Worklist → Viscous Liquids Handling → Mixing → pH Adjustment → Wait Time → Characterisation







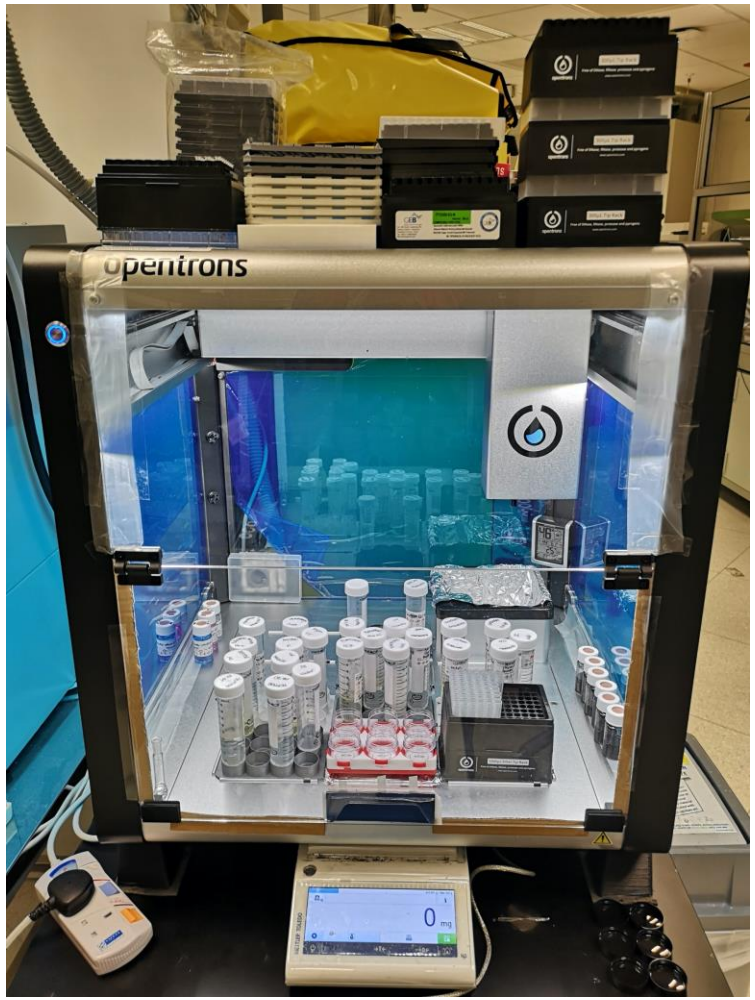
Aniket Chitre, Alexei Lapkin et. al. (2023)

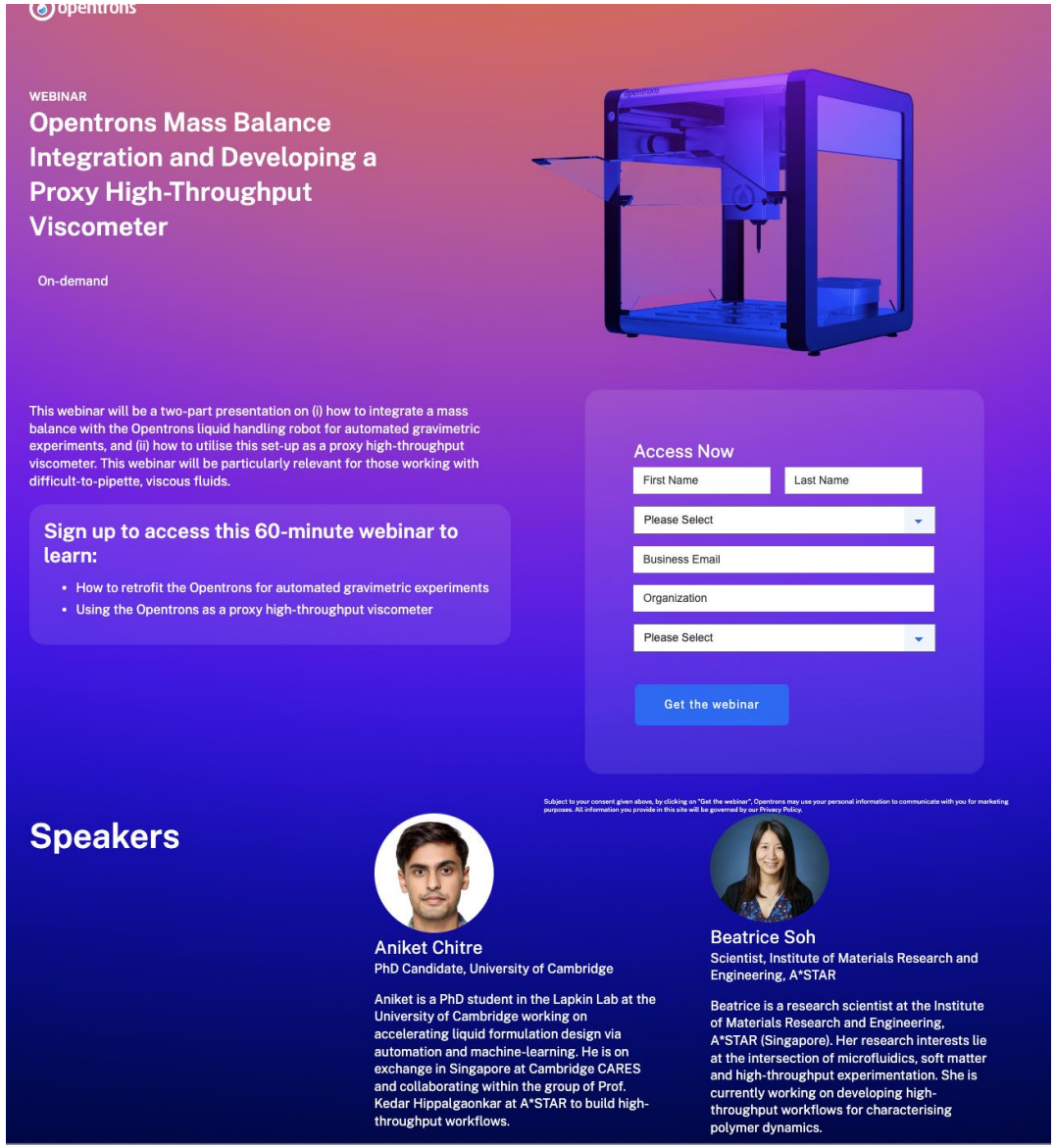
BASF We create chemistry

UNIVERSITY OF CAMBRIDGE

https://insights.opentrons.com/webinar-09-21-23-mass-balance-integration

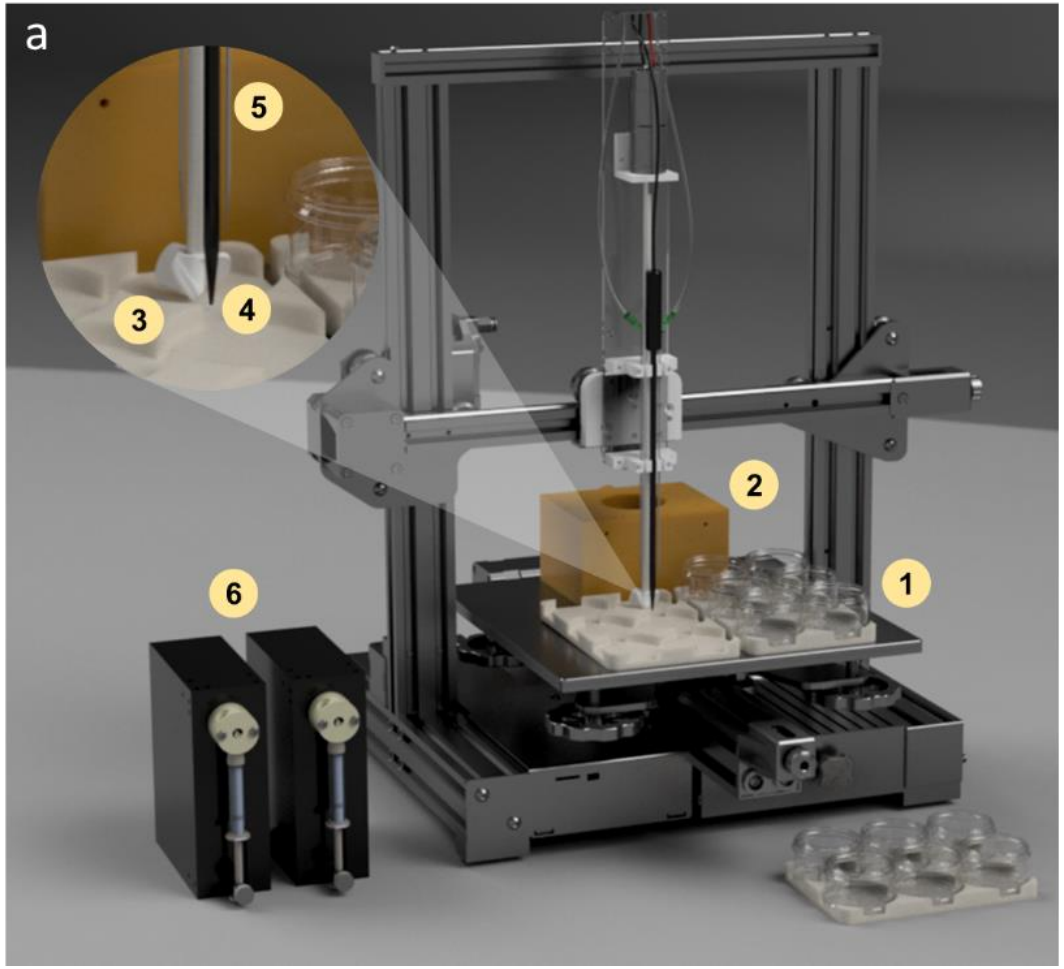
Viscous Liquids Handling

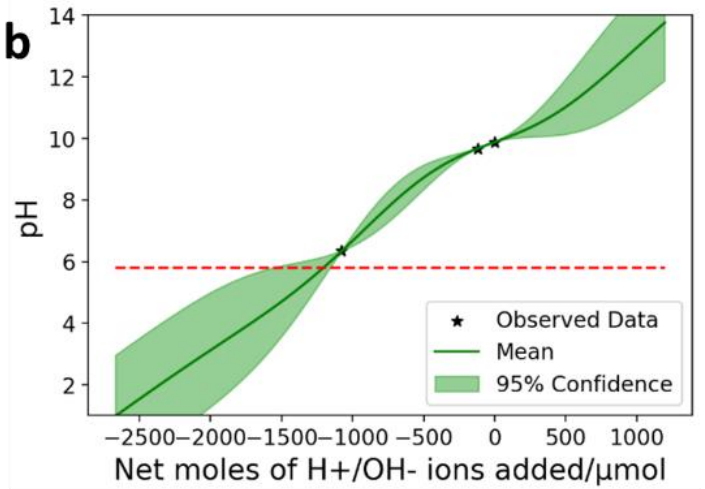


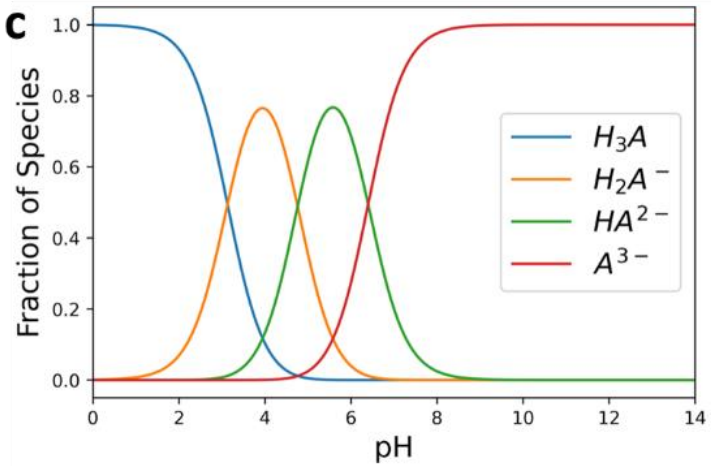


Aniket Chitre, Beatrice Soh, Kedar Hippalgaonkar et. al. RSC **Digital Discovery** (2023)

pHbot: Automated, ML-driven pH Adjustment of Viscous Formulations







10.26434/chemrxiv-2023-c46mv

Aniket Chitre, Jayce Cheng, Alexei Lapkin et. al. in review **Chemistry Methods** (2023)

phbot: Supplementary ~~Information~~ Instructions

10.26434/chemrxiv-2023-c46mv

pHBÖT

AMDM Design and Quality AMDM of Singapore

1 GENERAL INFORMATION

DESIGNER INSTITUTION
Aniket Chitre Cambridge Centre for Advanced Research and Education in Singapore
Jayce Cheng Institute of Materials Research and Engineering, A*STAR, Singapore

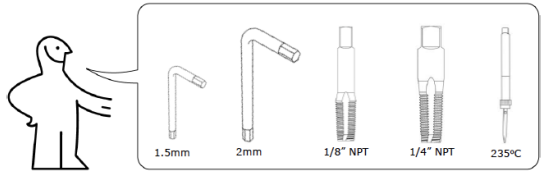
2 DESCRIPTION

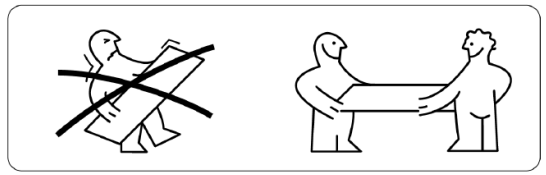
This is the assembly guide for the pHBOT: self-driving titration robot.

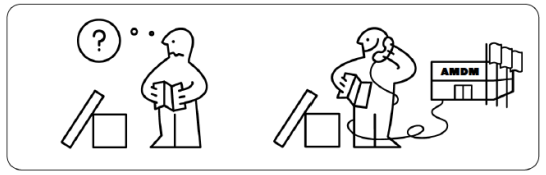
*Labware not included
*Wiring and tubing not shown

3 RESOURCES

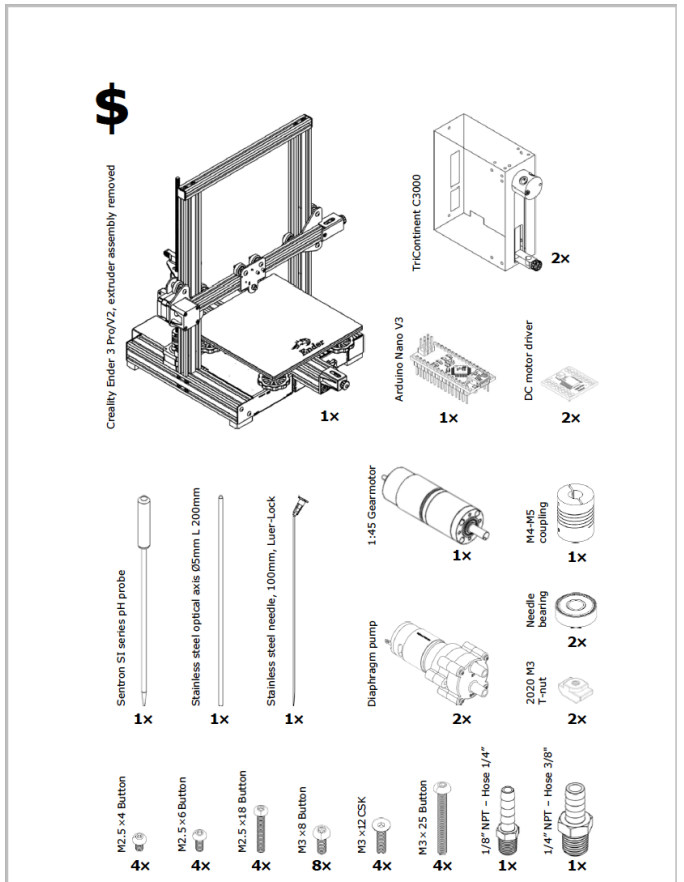
- Access to FDM 3D printing
- Access to SLA, DLP or equivalent resin 3D printing
- Basic soldering



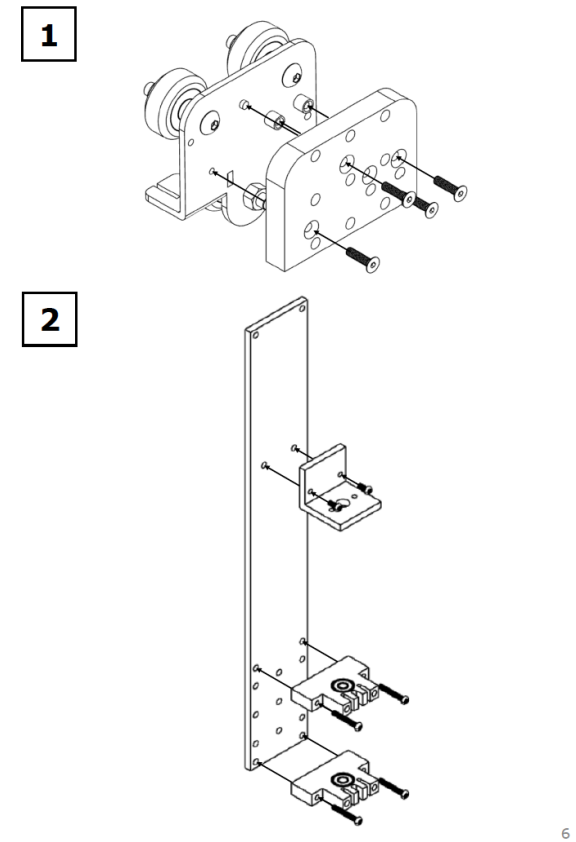




1



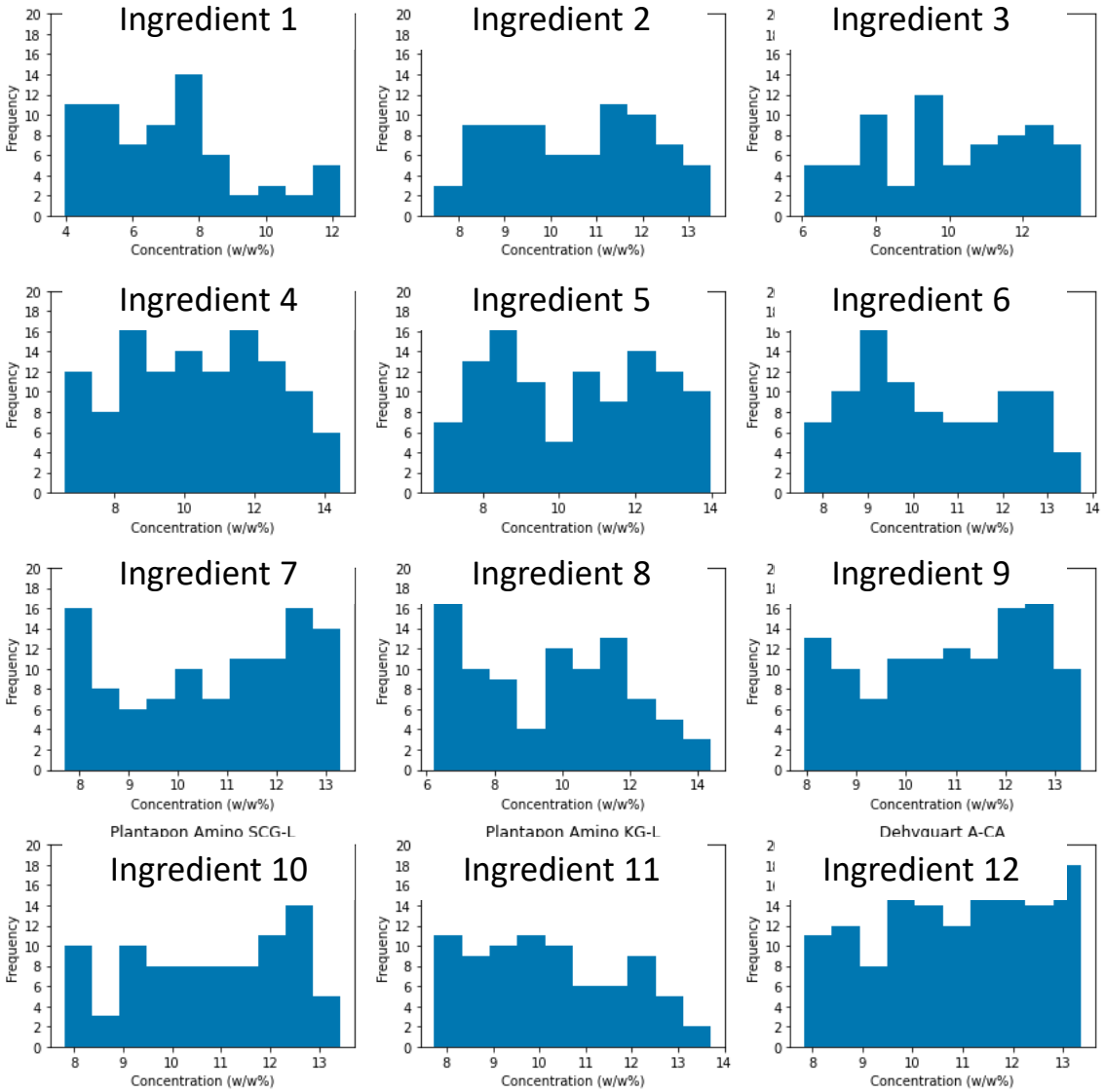
2

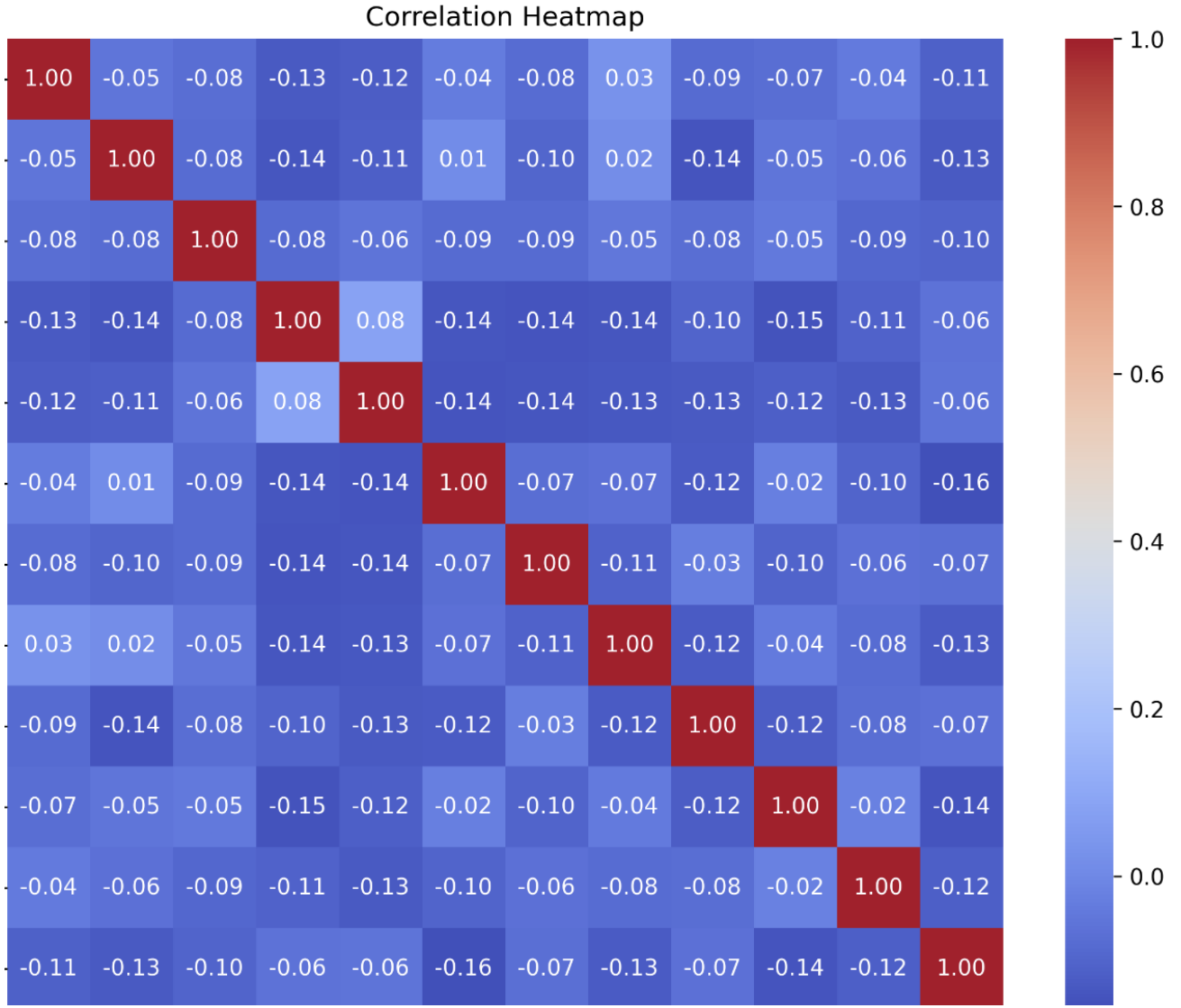


6

Aniket Chitre, Jayce Cheng, Kedar Hippalgaonkar, Alexei Lapkin et. al. **Chemistry Methods** (2023)

Effective Sampling – target is NOT optimization, but balanced dataset





Aniket Chitre, Kedar Hippalgaonkar, Alexei Lapkin et. al. **Chemistry Methods** (2023), **Nature Scientific Data** (2024)

AI for New Materials Discovery

Microscope, electrical probe

ender 'bots'

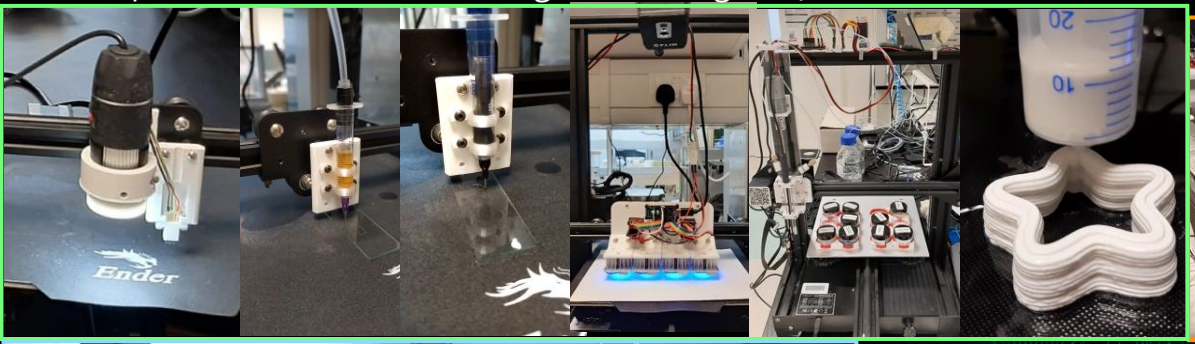
Hydrogel

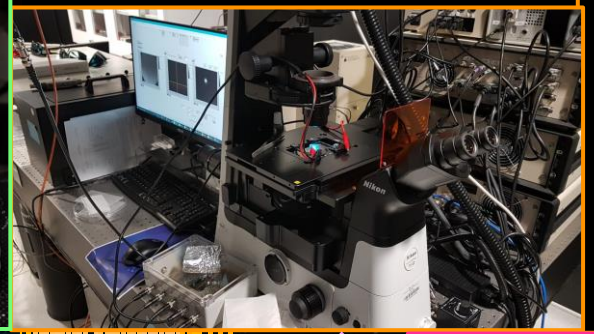
Ink Printing

UV curing

pH titration

Concrete





DNA & polymers

Automatic Electrokinetic Stretcher

Nature Comp Science **4**, 66 (2024)

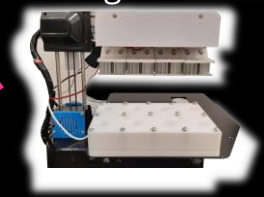
Sustainable Materials

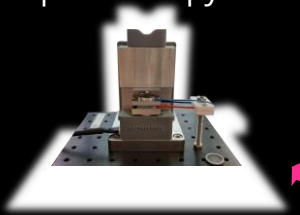
Battery Materials

Auto-Pipetter

Spin Coater

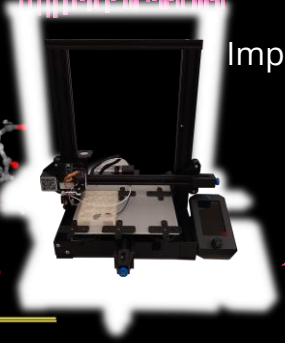
UV Curing



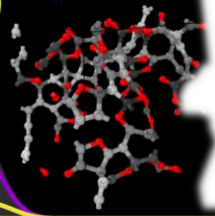


Dynamic Mechanical Analysis (Thermal)

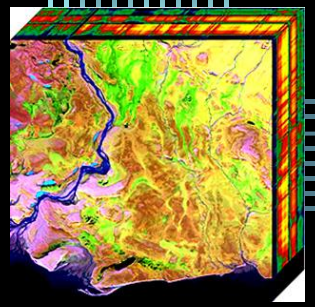
Electrochemical Impedance Spectroscopy



XPS

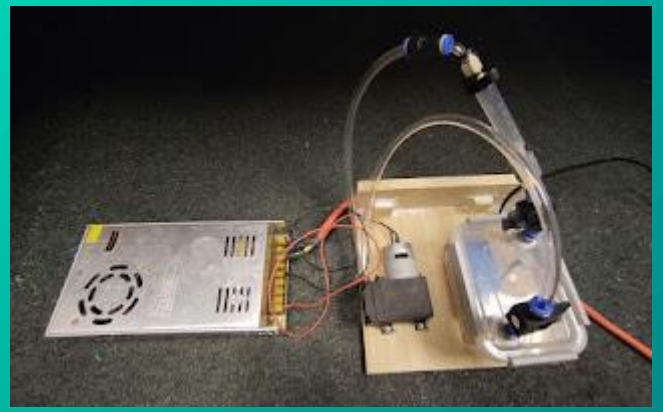


Hyperspectral Imaging



Example #4: Xinterra - carbon capture fabric

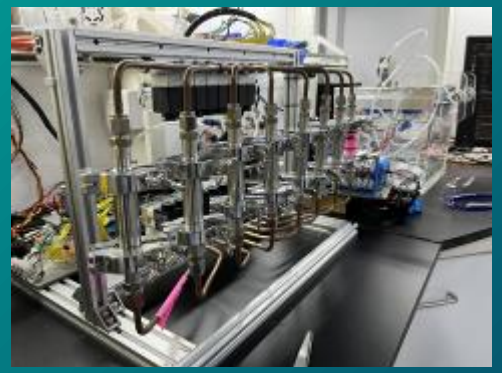
*A*STAR spinoff, technology licensed, IMRE staff/students co-founders*



3 months

2 prototypes

1.5 FTEs



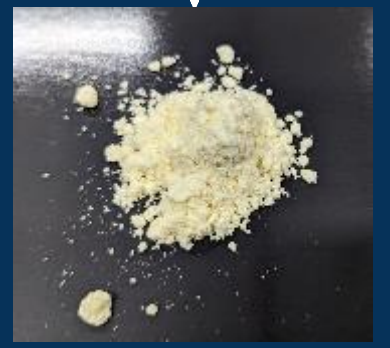
4 months

120 materials

3 FTEs

AI Round 1

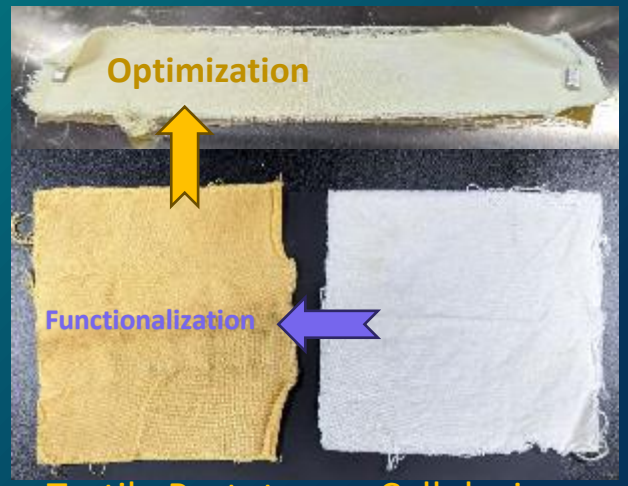
2 FTEs



Powdered cellulose

AI Round 2

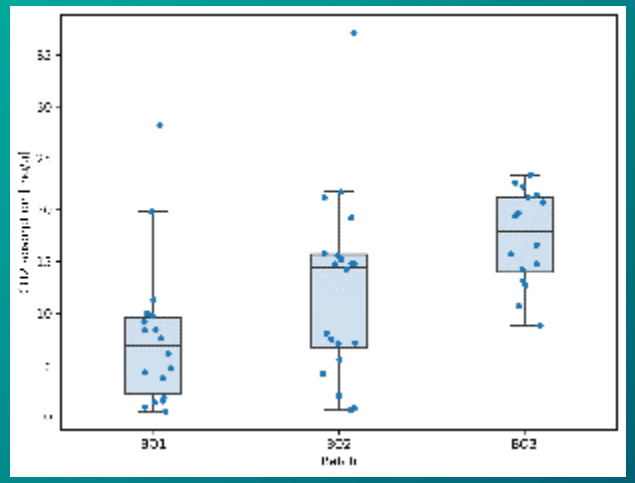
2 FTEs



Textile Prototypes: Cellulosics

AI Round 3

2 FTEs



AI driven optimization + IP Filing

4 months, TRL 2 to 5, now discussion with toll manufacturers.

Accelerated Catalyst Development Platform (US$8M, 36 months)

Characterisation

Detailed and High-Throughput Simulation

Institute of Materials Research and Engineering
IMRE

Institute of Sustainability for Chemicals, Energy and Environment
ISCE²

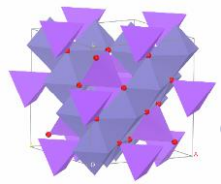
Institute of High Performance Computing
IHPC

HTE Synthesis

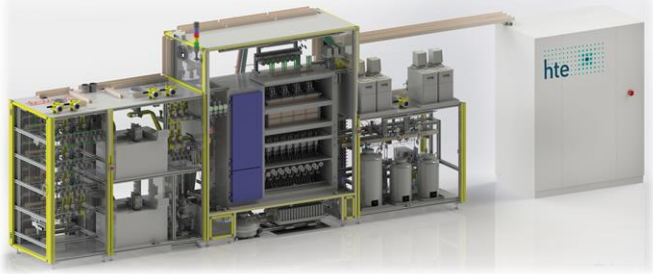
Novel/improved catalyst

High-Throughput Experiments

AI/ML



16-fold high throughput unit



CREATING GROWTH, ENHANCING LIVES

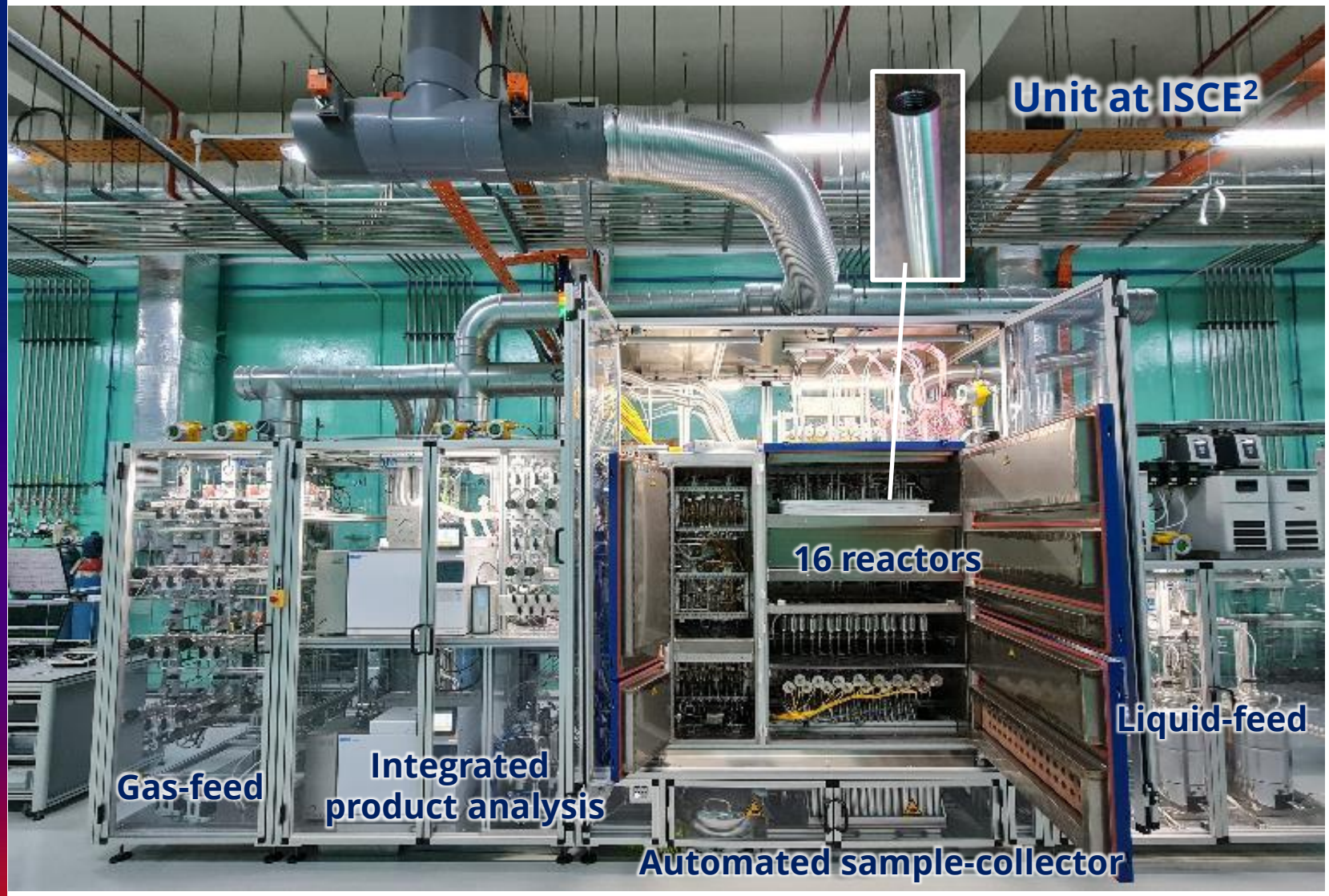
High-throughout Catalyst Synthesis

- Automated high-throughput catalyst synthesis system

- ✓ Up to 16 parallel catalyst syntheses
- ✓ Industrially widely used methods: **Impregnation, co-precipitation, sol-gel**
- ✓ In-built automated pH monitoring
- ✓ In-built post-synthesis drying
- ✓ Multiplies output by >5 times

High-throughout Catalyst Screening

- Automated high-throughput catalyst screening system



- ✓ 16 parallel reactor system
- ✓ Gas feed → Plug-flow
- ✓ Liquids feeds → Trickle-bed
- ✓ RT to 700°C
- ✓ Up to 200 bar
- ✓ Up to 8 ml catalyst scale
- ✓ Powder or shaped forms
- ✓ Online GC & offline analysis
- ✓ Multipoint T monitoring
- ✓ Multiplies output by >5 times

CREATING GROWTH, ENHANCING LIVES

Industry adoption of AI developed materials for CO_2 utilization

CREATING GROWTH, ENHANCING LIVES

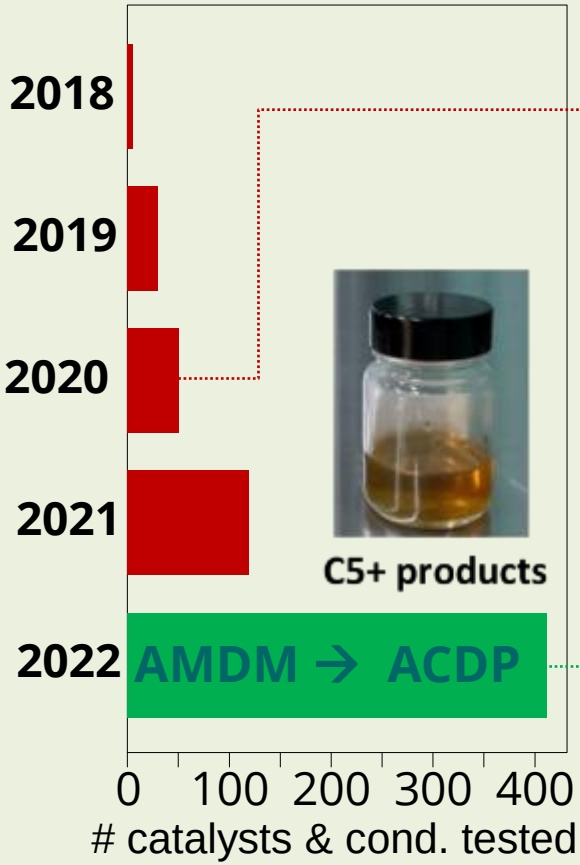
Traditional Development

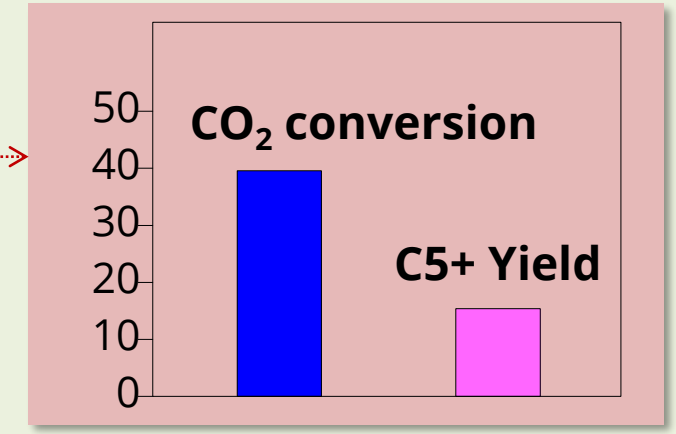
CO_2 methanation technology by conventional approach took more than 10 years

- **2011** — **TRL 1-4**
  Lab feasibility
- **2015** — **TRL 5-6**
  Bench& Field tests
  0.2-2 kg CO_2/day
- **2019** — **TRL 7-9**
  System test
  0.5 ton CO_2/day
- **2020** — **1st commercial order**
- **2022**
  24MT CO_2 capture/day
  8MT CH_4/day

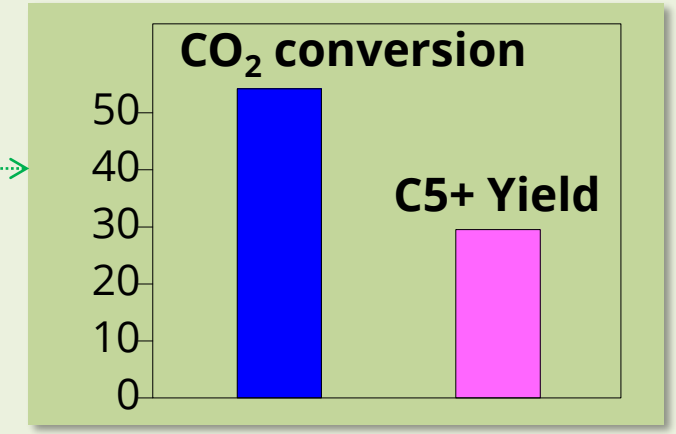
From 10 years down to 2 years!

CO_2 to SAF via ACDP embarks on TRL 5/6 within a year

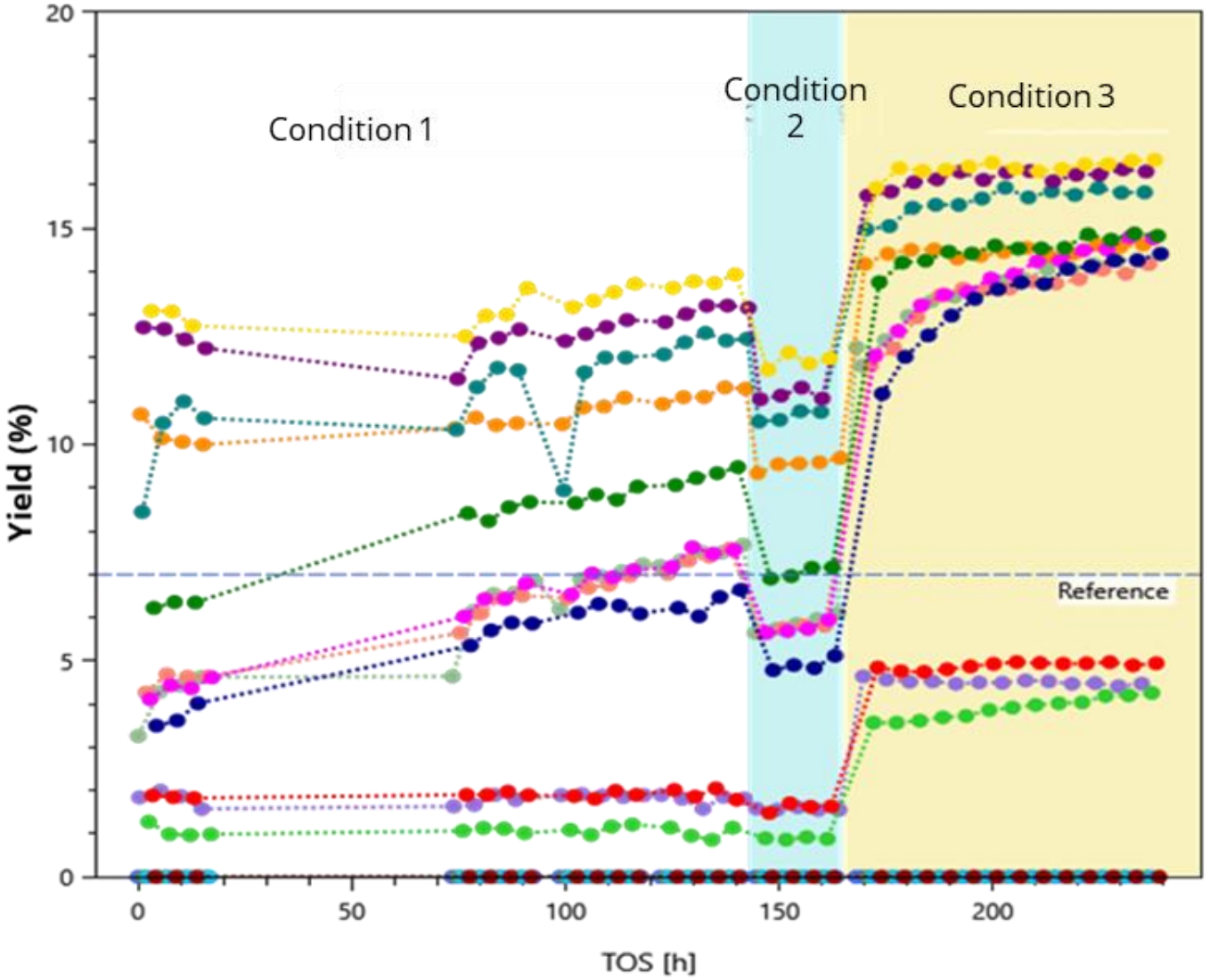




2x more yield via ACDP

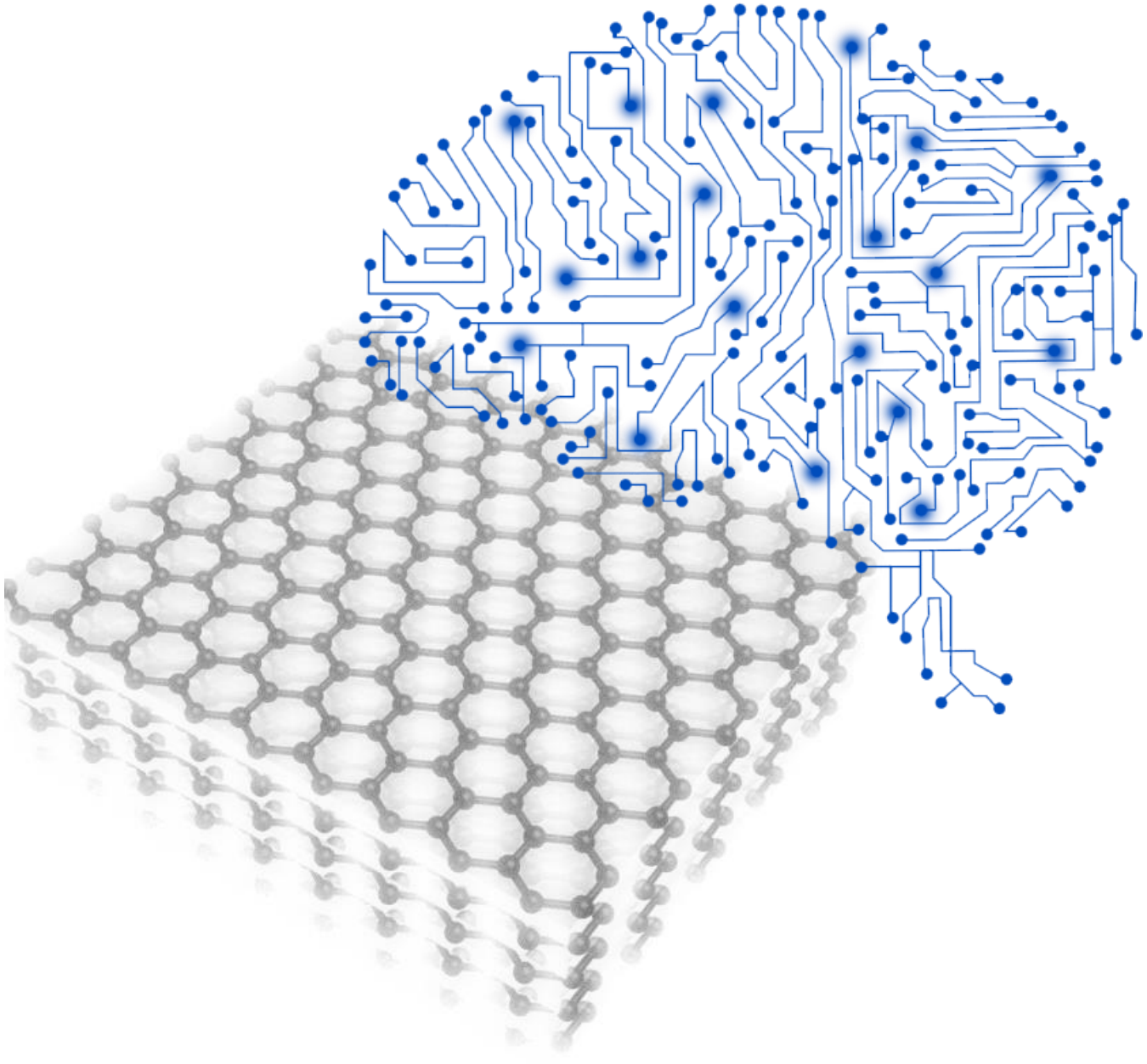


Parallel in-line monitoring of Fischer Tropsch reactions



- 16 catalysts are evaluated in HTE fixed bed reactor simultaneously
- 3 different reaction conditions are tested, over 200 hours Time on Stream
- In-line GC monitoring allows quantification of both gaseous and liquid products
- >10x acceleration in catalyst screening time

CREATING GROWTH, ENHANCING LIVES



What's next?

Generative Model for Inorganics:
MAT-GDT (Materials Generative Design and Testing Framework

Data Driven Design of functional materials

Discovering **new sustainable materials** with **desired performance** requires

(a) generative design and development of a **foundation AI** model,

(b) accelerated validation through high-throughput simulations and experiments (self-driving labs)

Language – (LLMs)
Chat GPT, BingChat etc.

Images – **Stable Diffusion**

Materials? – **Mat GDT!**

CREATING GROWTH, ENHANCING LIVES

Property-directed design

LLMs

2(i) Materials Knowledge
2(ii) Synthesis Recipes

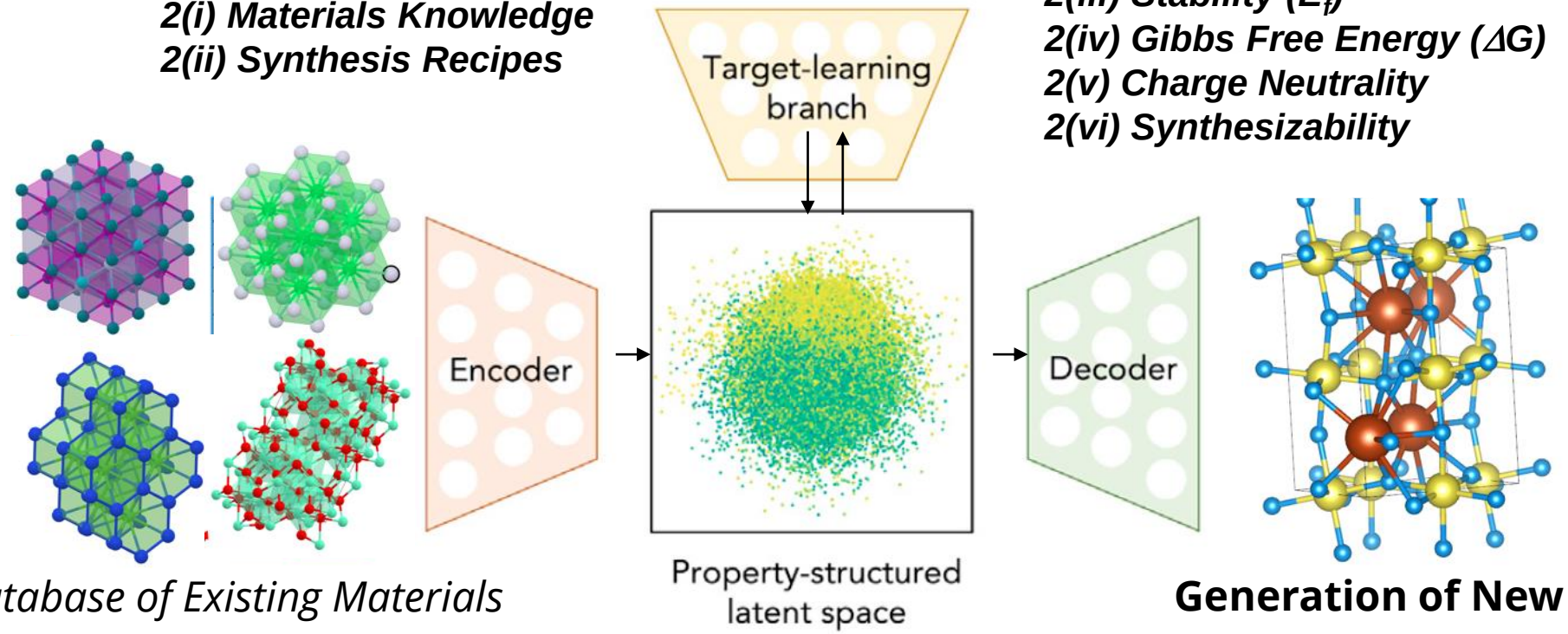
Material Properties (E_f, E_g…)

Ground State

2(iii) Stability (E_f)
2(iv) Gibbs Free Energy (ΔG)
2(v) Charge Neutrality
2(vi) Synthesizability

Applications

2(vii) electronic
2(viii) thermal
2(ix)–HER/OER, optical



Database of Existing Materials

Generation of New Materials

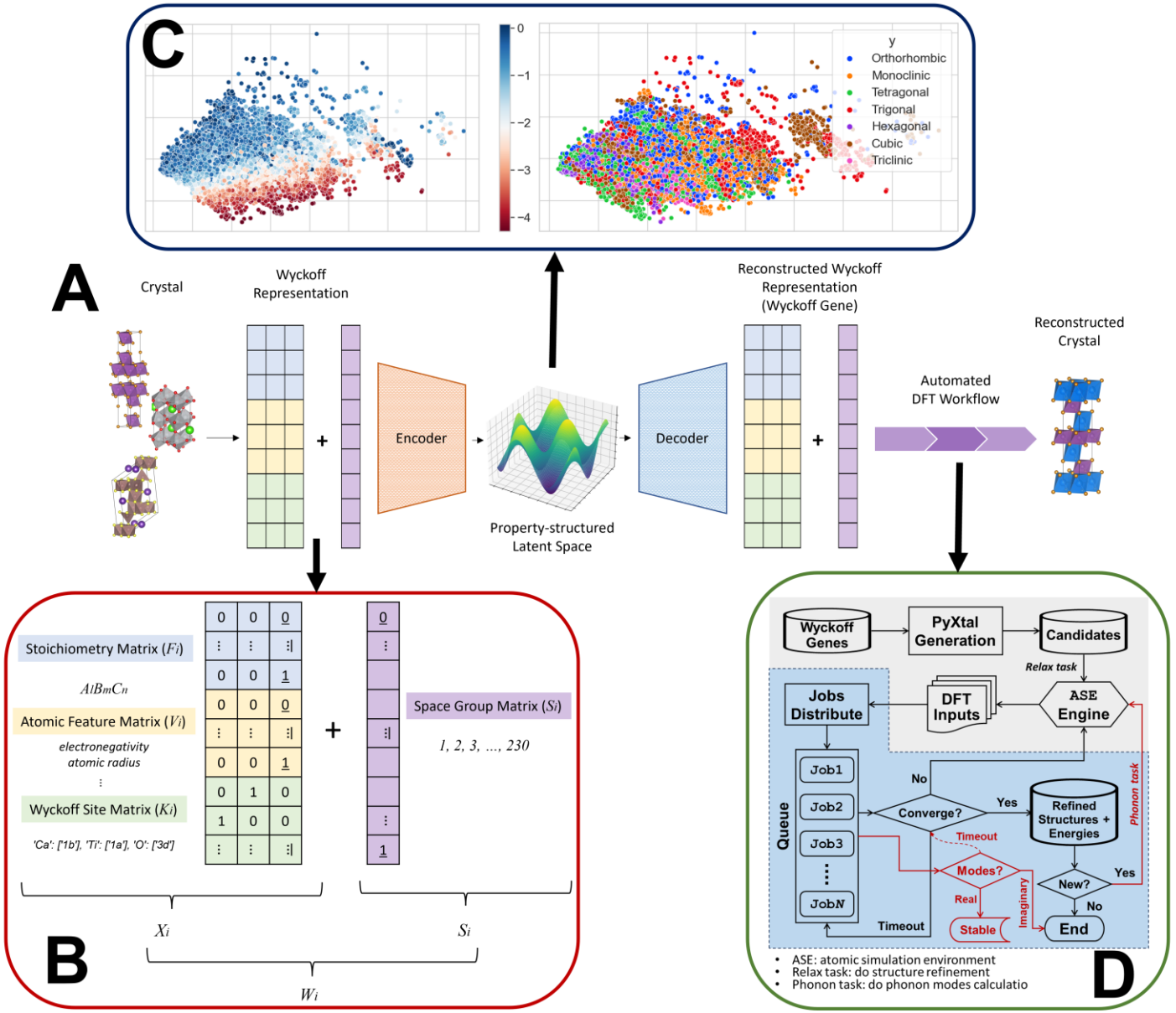
A. Inorganic Material Representation

B. Foundation AI Model Development

C. Automated Theoretical and Experimental Validation

R. Zhu et. al. https://doi.org/10.1016/j.matt.2024.05.042 ***Matter (2024)***

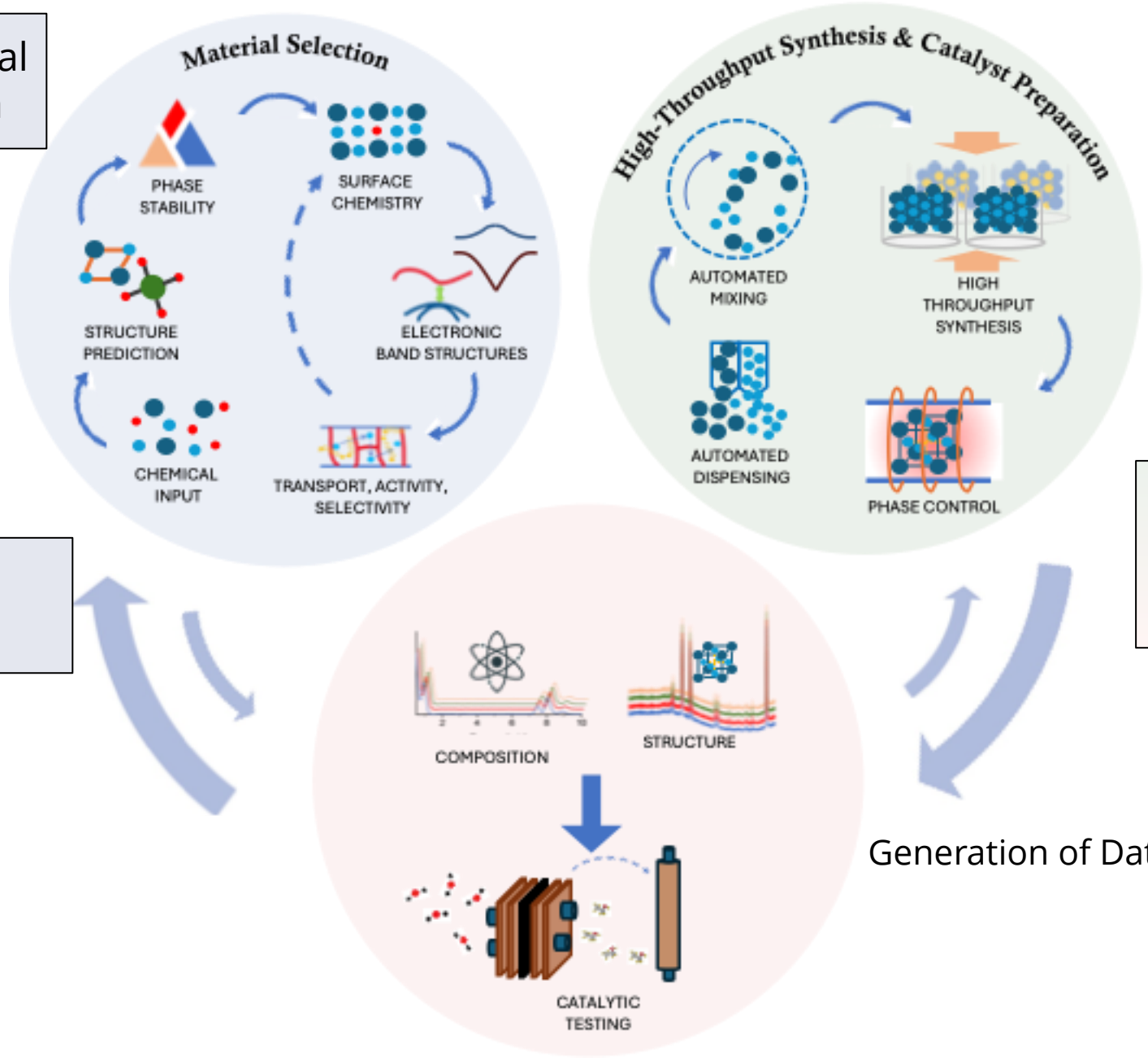
WyCryst – Wyckoff Inorganic Crystal Generator Framework



Zhu et. al. https://doi.org/10.1016/j.matt.2024.05.042 **Matter (2024)**

AI and high-throughput integrated catalyst design

Inorganic Material Representation

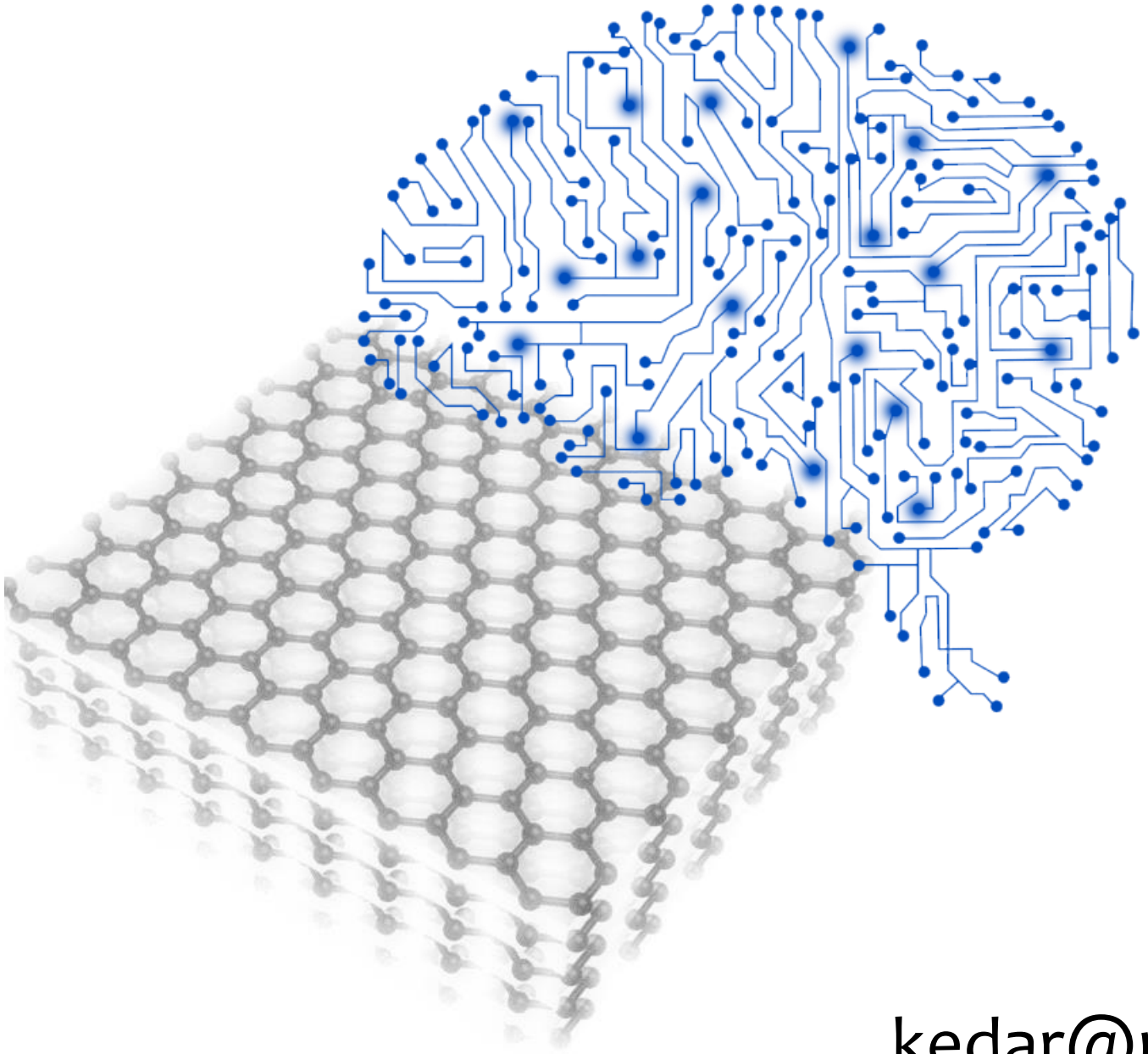


AI Model Development

High-Throughput Theoretical and Experimental Validation

Generation of Datasets

CREATING GROWTH, ENHANCING LIVES



Conclusions

- AI-integrated Material—by-design approach allows for property directed searches.
- Databases for functional properties need to be generated
- DFT and Rapid Experimental Validation is necessary

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