Conversion of Wet Waste to Fuel and Value-Added Products using Hydrothermal Carbonization

HTC of food waste: current status; How to run a DOE for HTC

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Part 5- Case study of HTC of food waste

Session 1: Review of the behaviour of food waste HTC

Session 2: Introduction to factorial design of experiments

Session 3: Case study of food waste HTC outputs

Session 1- Review of developments in food waste HTC

This session will cover:

- Proximate and ultimate analysis
- Determination of calorific value
- Analysis of inorganics in hydrochar

Session 2- Introduction to factorial design of experiments

This session will cover:

- Analysis of combustion properties of hydrochars
- Analysis of ash chemistry
- Agronomic analysis (CEC, humic acids, germination tests)
- Environmental analysis (PAH, leaching)

Session 3- Case study of food waste HTC outputs

This session will cover:

- X-Ray photoelectron spectroscopy
- Infra Red analysis
- Gas adsorption analysis

Session 1
Review of HTC of food Waste

Session 2-Hydrothermal carbonisation for food waste valorisation

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Increasing pattern in number of research works on HTC of FW

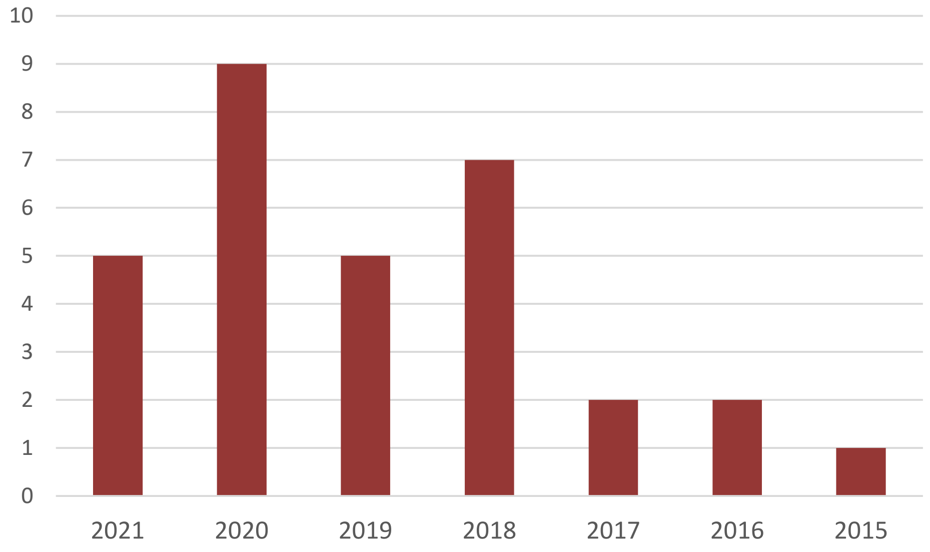


Fig. 1 - Published papers of FW-HTC in recent years

- Pre-consumer food waste (industrial waste)
- Post-consumer food waste (household waste)
- Organic fraction of municipal solid waste (OFMSW)

Session 2- Research on utilisation of FW-HTC

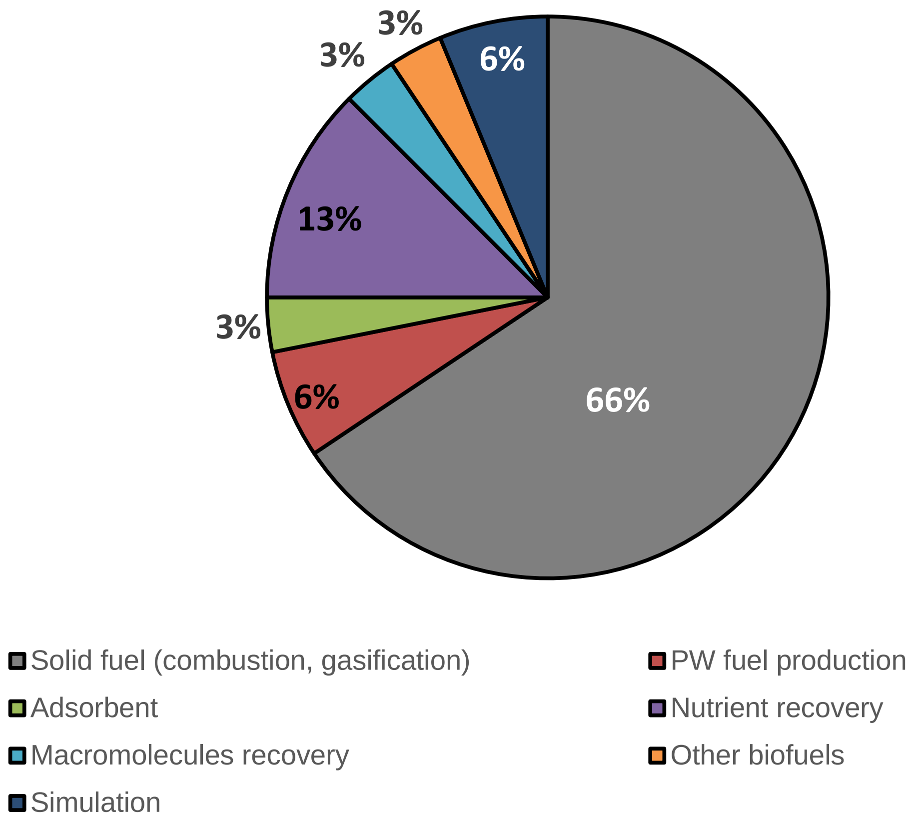


Fig. 2 - Pie chart of FW-HTC utilisation

- Solid fuel is the main utilisation of FW-HTC
- Few other utilisation are explored for FW = research opportunities
- 18% of published papers include process evaluation

Session 2-Composition of FW hydrochar

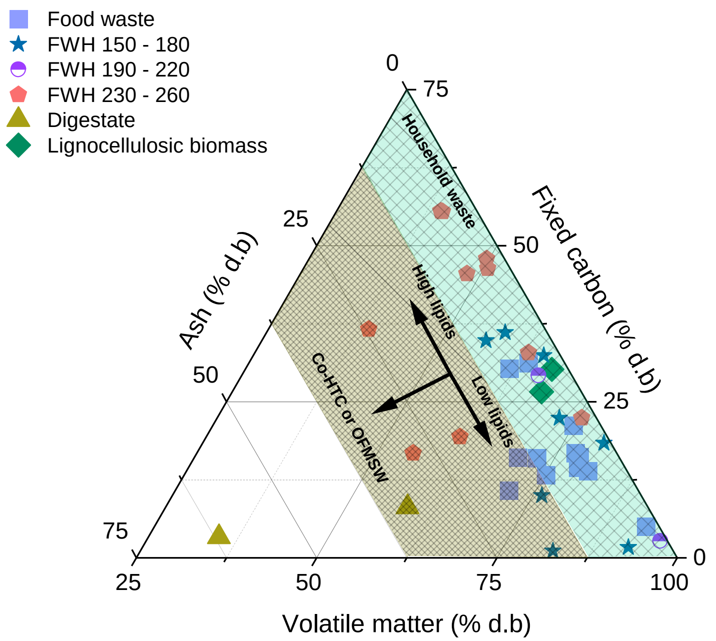


Fig. 3 - Ternary plot of proximate composition of FW hydrochar

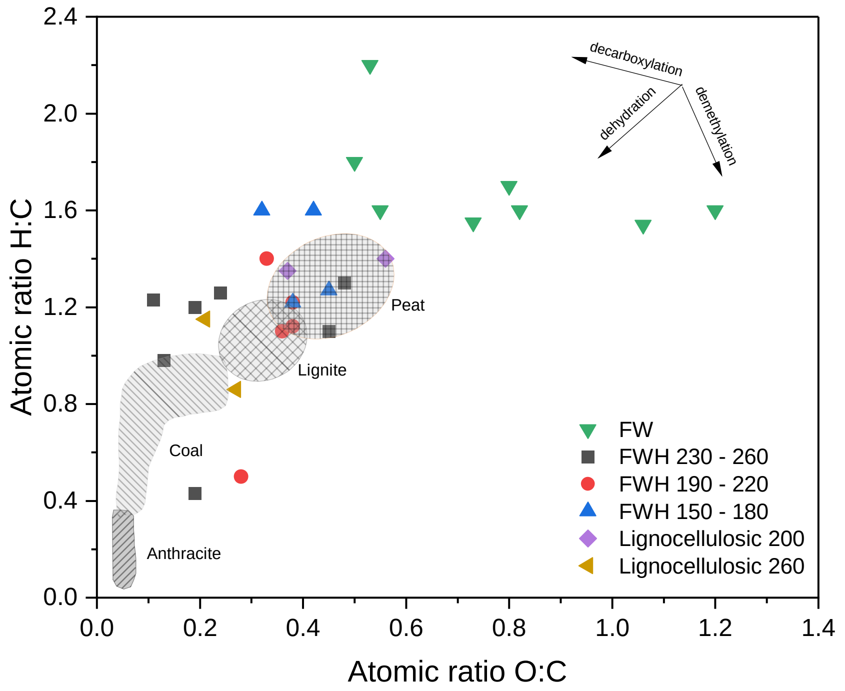


Fig. 4 - Van Krevelen diagram of FW hydrochar

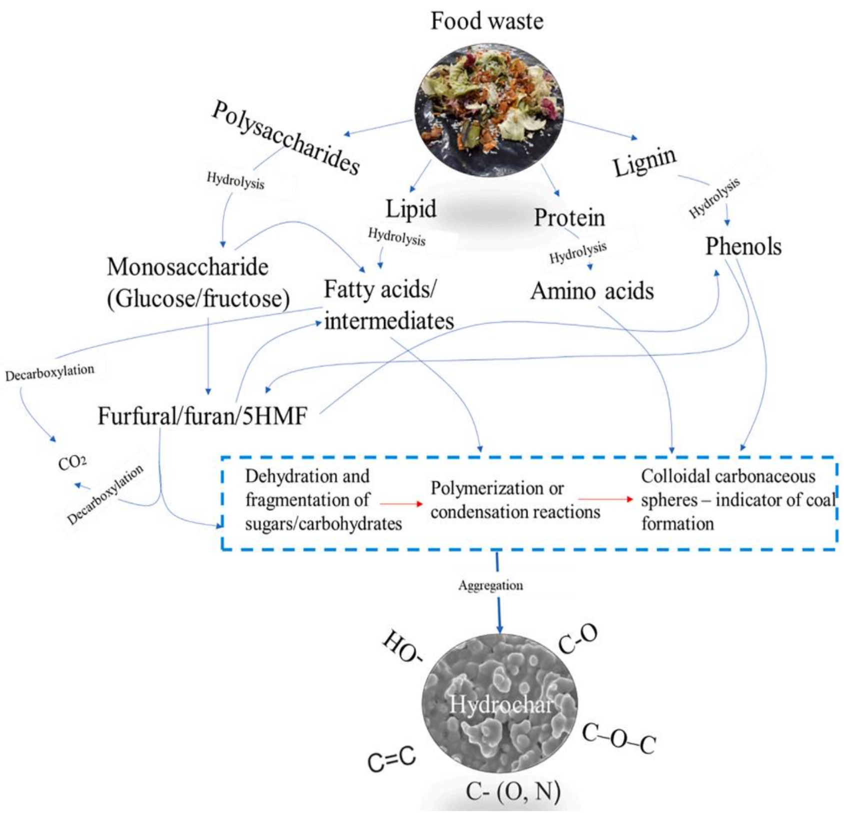
Session 2- Composition of FW hydrochar

FW hydrochar solid fuel responses

Feedstock	Temperature	Yield (%)	HHV (MJ/kg)	EY (%)	ED	Reference
Mixed post-consumer FW	220 - 260 °C	-	19.55 - 29.77	26.95 -23.57	1.85 - 2.82	Sharma et al., 2021
Pre-consumer FW	180 - 220 °C	37 - 56	19.60 - 25.36	50 - 71	1.13 - 1.47	Wilks et al., 2021
Mixed post-consumer FW	180 - 250 °C	39.5 - 72.5	19.5 - 25.6	-	-	Ischia et al., 2021
Mixed post-consumer FW	180 - 250 °C	50.1 - 40.9	22.4 - 26.7	65.5 – 63.7	1.3 - 1.56	Picone et al., 2021
Mixed post-consumer FW	220 - 260 °C	59.83 - 45.27	24.37 – 27.64	59.98 – 45.29	1 -1	Sharma and Dubey. 2020
Mixed post-consumer FW	160 - 200 °C	52 - 58.4	23.3 - 29.6	-	-	Gupta et al., 2020
Mixed post-consumer FW	180 – 280 °C	30.5 – 27.5	23.5 – 29.6	37.4 – 42.4	-	Mazumder et al., 2020
Mixed post-consumer FW	175 - 250 °C	40 - 44	21.6 - 26.7	-	1.18 - 1.46	Akarsu et al., 2019
Mixed post-consumer FW	200 - 260 °C	75 – 68.5	30.45 - 33.08	-	1.21 - 1.31	McGaughy and Reza, 2018
Mixed post-consumer FW	200 - 250 °C	23.8 - 28	31	-	1.83 - 1.95	Saqib et al., 2018

Although is there is increasing data on FW-HTC, optimisation studies are required to bring insight and application to the process.

Session 2- Optimization of hydrothermal carbonisation



Sharma et al., 2021

- Multiple simultaneous reactions of HTC
- Feedstock dependant
- Different biomolecules in food waste
- Different proportions dependent of feedstock source

Complicate the generation of a general model of HTC and numerical optimization.

DOEs are an useful option to develop empirical models and optimization.

Session 2-DOE in HTC

DOE pros:

- Less experimental runs
- Generation of empirical model
- Significance test of process factors
- Optimisation

However:

- Most of HTC research is has been ‘one variable at a time’ (Traddler et al., 2018)
- Although DOE for HTC are gaining popularity, majority of the studies are still focusing on the same responses (solid yield, HHV).
- Few optimisation attempts, mainly single responses

Session 2-References

Type of DEO	Feedstock	Variables	Responses	Optimized conditions	Optimized responses	Reference
2-level factorial with center points	Microalgae	T, RT, SL	SY, CY	-	-	Heilmann et al., 2010
Box-Behnken frational	Digested mail silage	T, RT, pH	Carbon content, CY	-	-	Mumme et al., 2011
CCD	Olive stone	T, RT, SL	SY, HHV	-	-	Alvarez-Murillo et al., 2015
CCRD	Sewage sludge	T, RT	SY, HHV, EY and ED	180/60 and 200/30	carbon recovery in liquid	Danso-Boateng et al., 2015
	Lignocellulosc	T, RT, SL	SY, ED and EY	-	-	Makela et al., 2015
CCD	Palm shell	T, RT, SL	SY	-	-	Nizamuddin et al., 2016
	Coffe husk	T, RT, SL	SY, Surface area	210/243/3.4:1	33.3 m^2/g	Ronix et al., 2017
CCD	Shrimp waste	T, RT	SY	180/120		Kannan et al., 2018
CCD	AD digestate	T, Rt, pH	C, P and N recovery	165/500/3.5	36 %SY, 0.8 O/C difference	Stutzenstein et al., 2018
CCD	Bamboo	T, Rt, HCl	SY, O/C ratios Levulinic acid	160/3h/0.37M	9.46% Levulic acid	Sweygers et al., 2018
CCD	Digested Sewage sludge	T, Rt, pH	Dewaterability and P release	170/1.93pH	48% SY, 70% P release	Luhman and Wirth, 2020
CCD	Date stone	T, Rt, catalyst dose	SY, C retention	200/120/20mg	59.71%SY, 75.84% C	Quadrihi et al., 2021
Box-Behnken	Bark	T, Rt, Stirring speed	SY, HHV	180/4h/600rpm	69.89%SY, 18.59 MJ/kg	Sultana et al., 2021

HTC is a technology with known trade-offs, hence multiple variable and multiple responses are required for optimisation.

Session 2

Design of Experiments (DoE)

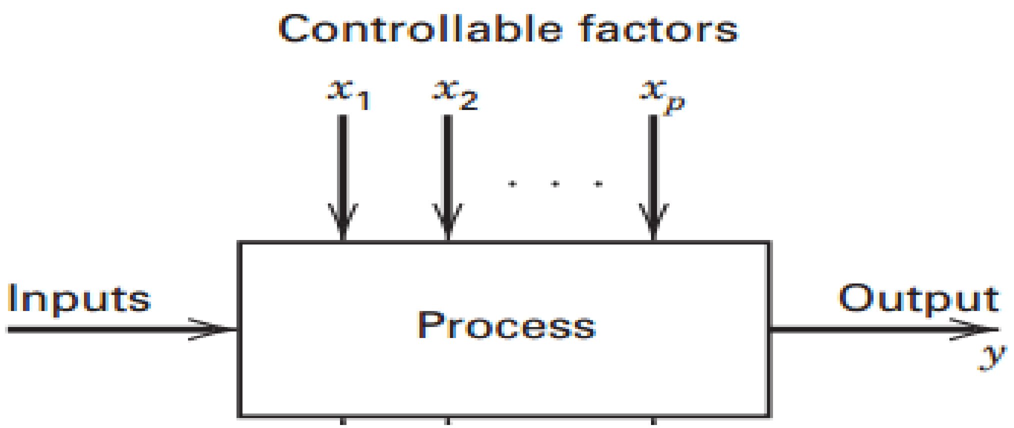
Basic concepts and applications

Session - Basis for Design of Experiments

This session will cover:

- ❑ Why using design of experiments?
- ❑ Factorial design
- ❑ Interactions and model validation
- ❑ Response surface
- ❑ Optimisation by desirability function

Objective of an experiment



It can be determined:

1. Most influential variables on the response *y*

2. Where to set the influential *x*'s so that *y* is almost always near the desired nominal value

3. Where to set the influential *x*'s so that variability in *y* is small

OFAT Vs DOE

Strategy for experimentation → One factor → Several factors

One factor-at-a-time (OFAT)

Select a baseline set of levels for each factor

Successively varying each factor over its range with the other factors held constant at the baseline level.

Time consuming

Does not consider any possible **interaction** between the factors.

Design of experiments (DOE)

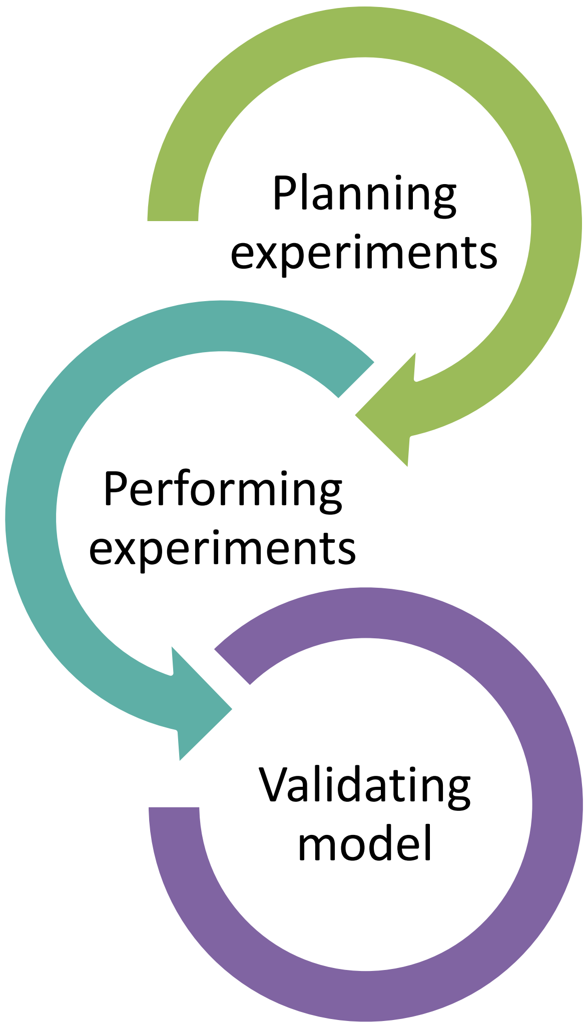
The correct approach to dealing with several factors is to conduct a **factorial** experiment.

This is an experimental strategy in which factors are varied *together*, instead of one at a time.

What is a DoE?

Design of Experiments

- DoE is a tool for studying the behaviour of a system
- Goal of DoE: Reduce experimental effort and increase quality of information



Applications of experimental design

1. Improved process yields

2. Reduced variability and closer conformance to nominal or target requirements

3. Reduced development time

4. Reduced overall costs.

5. Evaluation and comparison of basic design configurations

6. Evaluation of material alternatives

7. Selection of design parameters so that the product will work well under a wide variety of field conditions, that is, so that the product is **robust**

8. Determination of key product design parameters that impact product performance

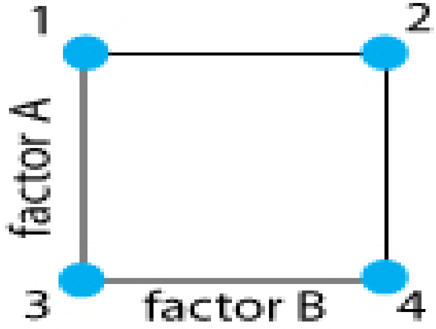
9. Formulation of new products.

Session 2- Steps for DoE

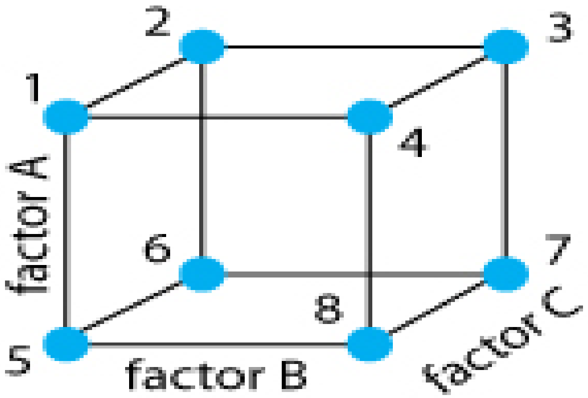
1. **State objective:** Needs to be clearly stated
2. **Choose response:** to increase understanding of mechanisms and physical laws involved in the problem
3. **Choose factors and levels:** A factor is a variable studied in the experiment.
4. **Choose experimental plan:** Crucial step for the success of the DoE
5. **Perform the experiment:** Use the DoE planning matrix
6. **Analyse the data:** Raw data analysis and model fitting
7. **Draw conclusions and make recommendations:** Conclusions should refer back to the stated objectives and should include the important factors. Also is useful to provide follow-up experiments.

Session 1 – Factorial design

- The effect of a factor, also known as main effect, is defined as the response change due to variations in the level of the factor.
- The levels are often stated as low (-1) and high (1).
- The treatment, trial or run is the combination of factor levels
- The planning matrix state the conditions for the experiments



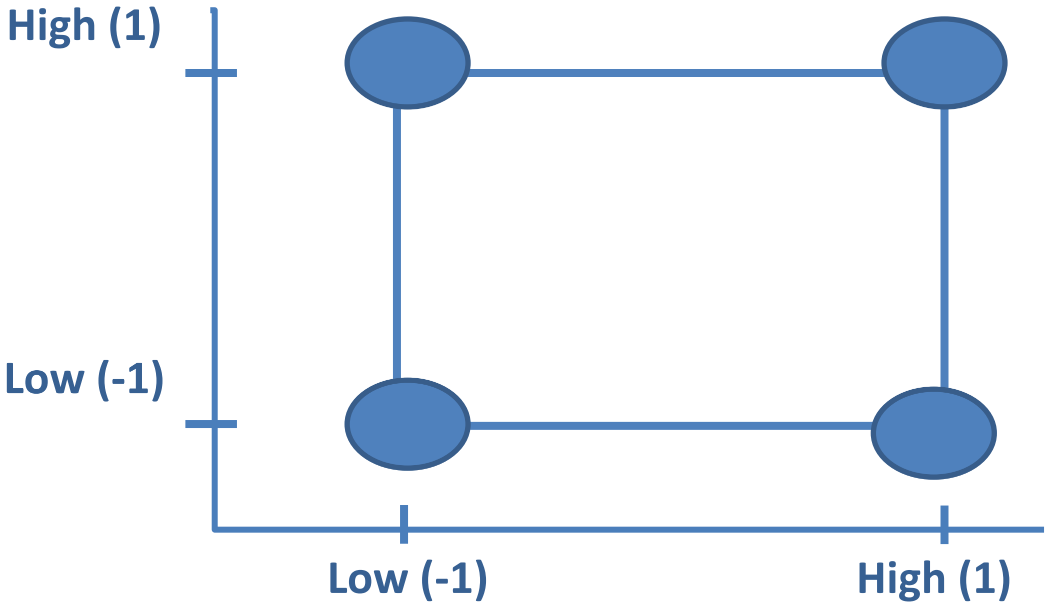
	factor levels	
trial	A	B
1	+	−
2	+	+
3	−	−
4	−	+



	factor levels		
trial	A	B	C
1	+	−	−
2	+	−	+
3	+	+	+
4	+	+	−
5	−	−	−
6	−	−	+
7	−	+	+
8	−	+	−

Session I – Factorial design

- Linear regression is used for fitting models to the experimental data
- ANOVA evaluates the accuracy of the model and the significance of the factors and interactions



2-factor factorial design

Linear regression model representation

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_{12} X_1 X_2$$

Y Response variable

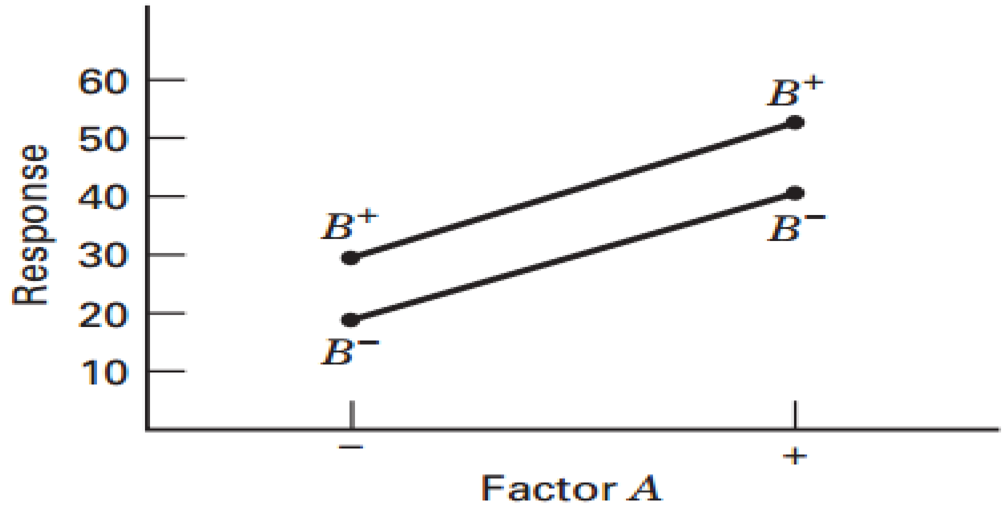
X Factor

β Coefficient

Session I –Factorial regression

Main effects

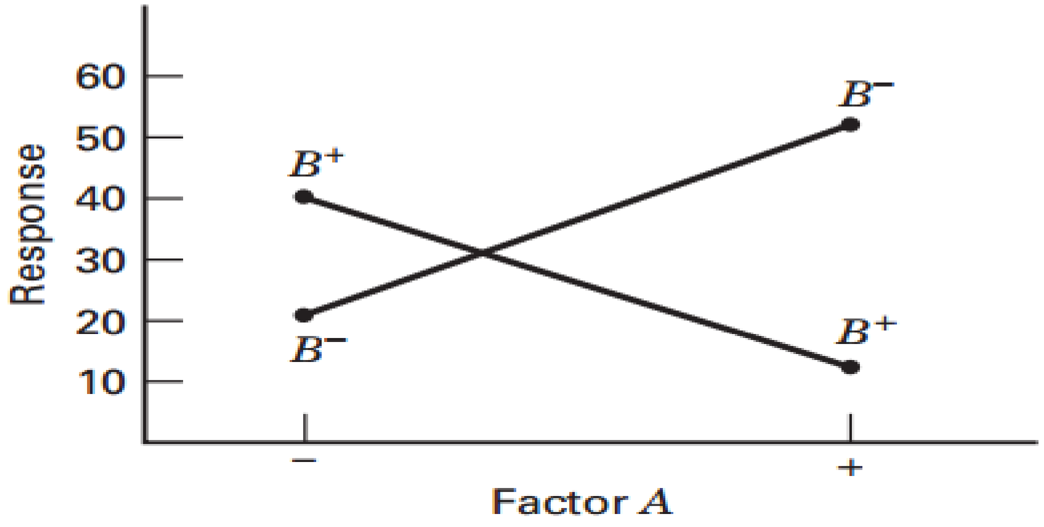
$$Y = \beta_0 + \beta_A A + \beta_B B$$



■ FIGURE 5.3 A factorial experiment without interaction

Interactions

$$Y = \beta_0 + \beta_A A + \beta_B B + \beta_{AB} AB$$



■ FIGURE 5.4 A factorial experiment with interaction

Session 1 – Factorial regression

Model validation

$$H_0 : \beta_1 = \beta_1 = \beta_2 \ldots = \beta_k = 0$$

$$H_1 : \beta_j \neq 0 \; for \; at \; least \; one \; j$$

Table 3
ANOVA of the final model. The tabulated values have been rounded.

Parameter	Degrees of freedom (df)	Sum of squares (SS)	Mean square (MS)	F-value	p-value
Total corrected	10	2.0			
Model	3	1.8	0.61	30.4	< 0.01
Residual	7	0.14	0.02		
Lack of fit	5	0.09	0.02	0.62	0.71
Pure error	2	0.06	0.03		

❑ Validation of the coefficients

Remove coefficients non statistically significant (p>0.05)

❑ Model application

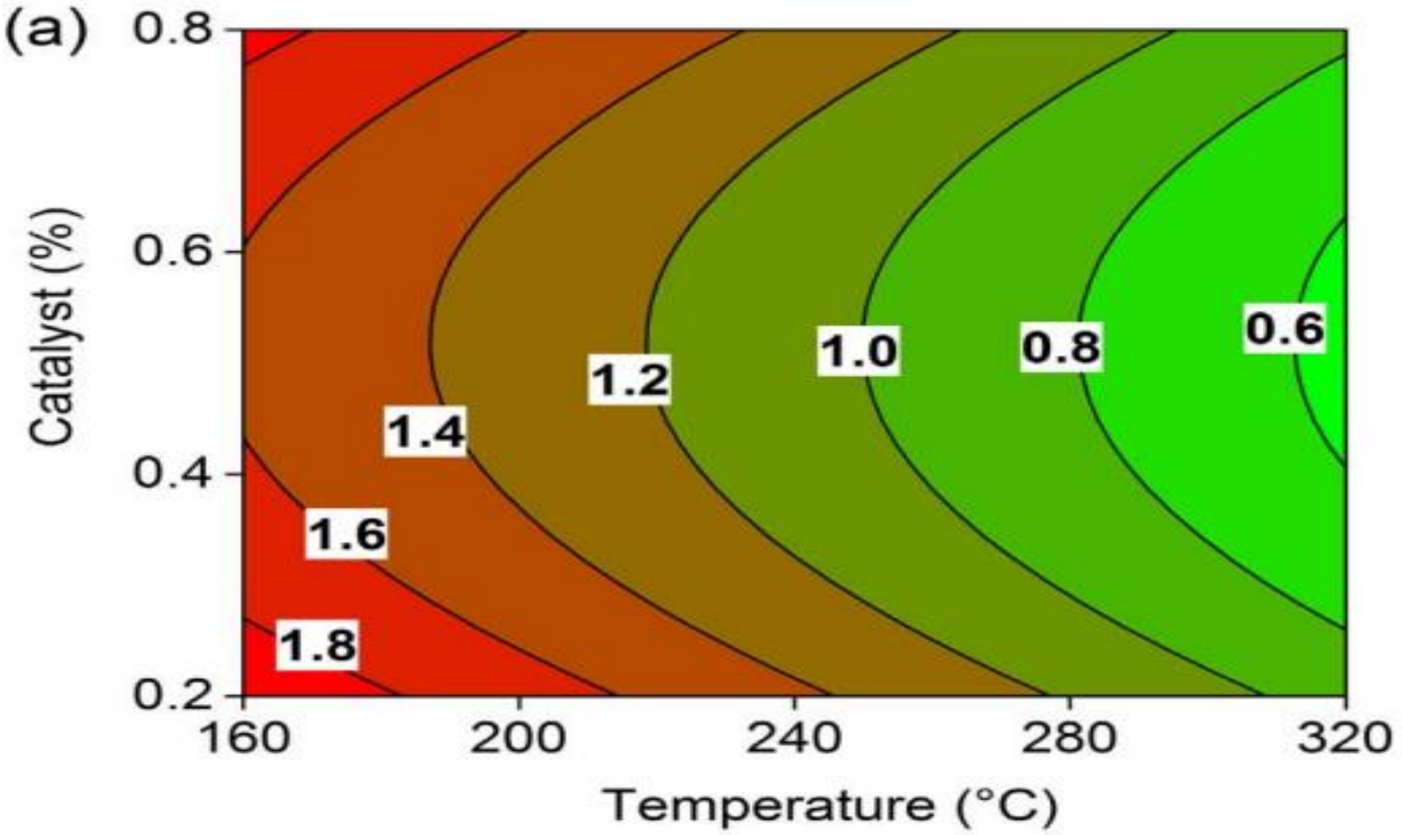
The obtained model can be use for predicting novel observations within the original design range.

Session 2- Design of Experiments

Visualisation of the optimal location

Graph of contours

- ❑ Evaluates 2 factors
- ❑ X and Y axis
- ❑ Response for maximum and minimum values

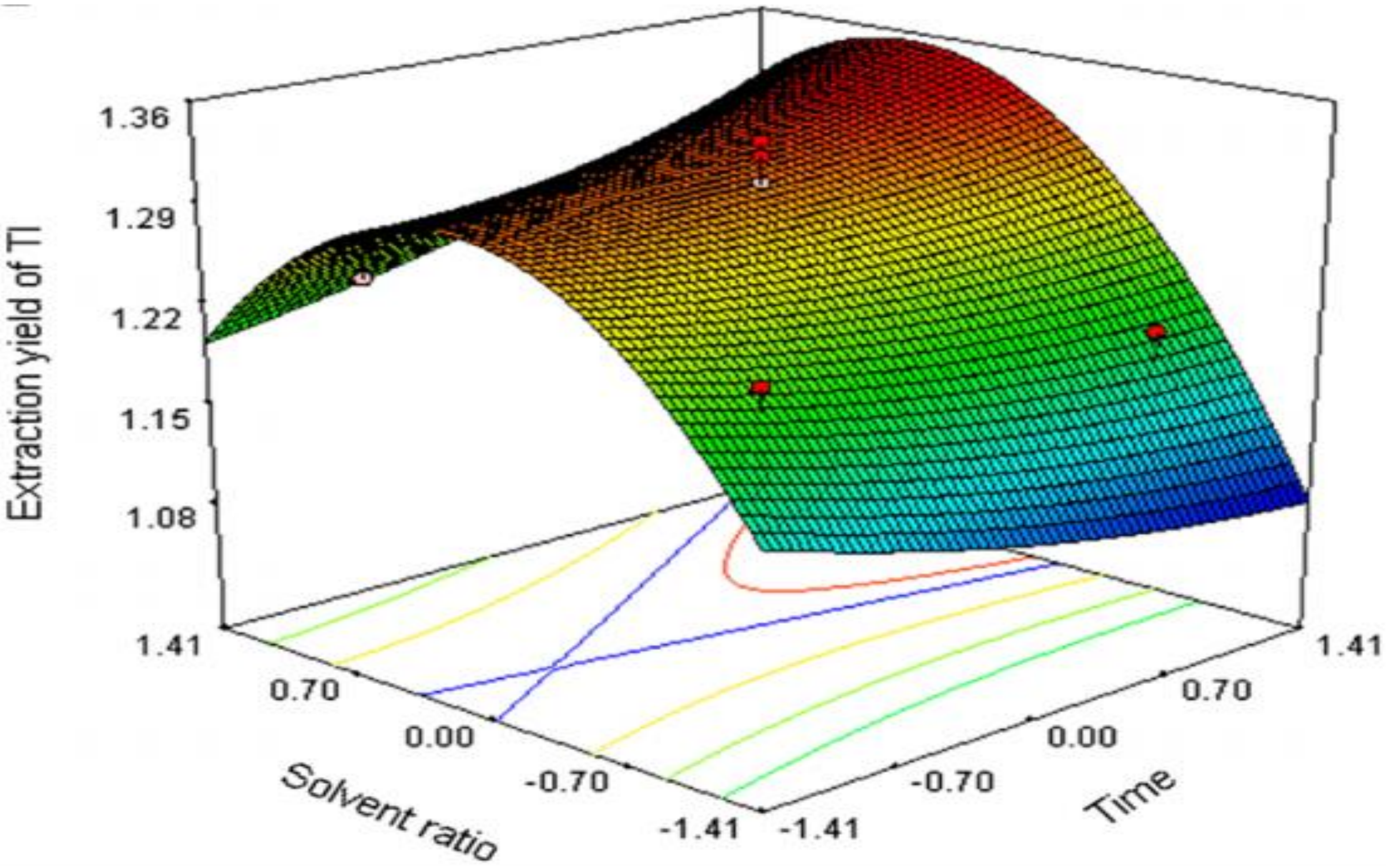


Session 2- Design of Experiments

Visualisation of the optimal location

3-Dimensional response surface

- ❑ Evaluates 3 factors



Session 2- Design of Experiments

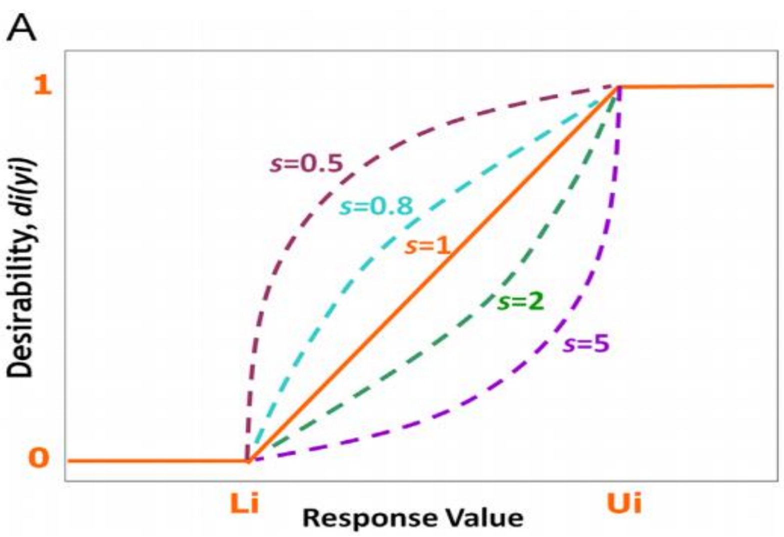
Multiple response optimisation

Graphical optimisation	Desirability function (D)
Only useful when 1 or 2 responses are considered	Useful for multiple responses

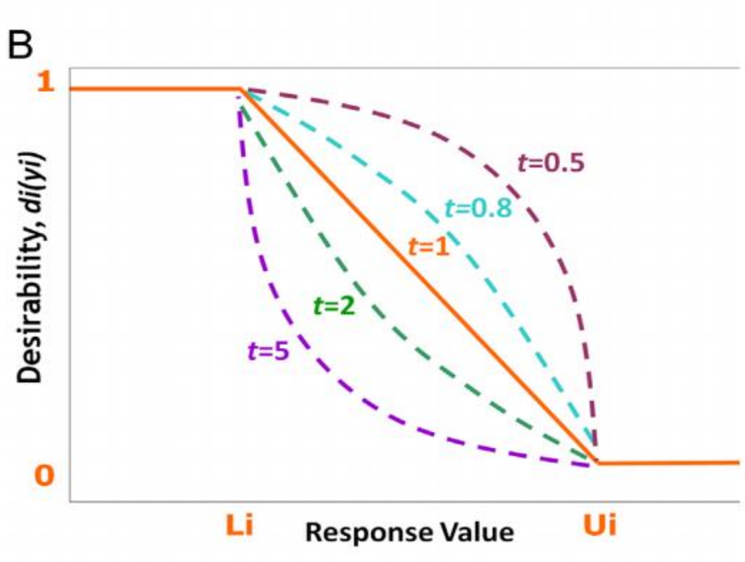
Desirability function (D)

- D considers several responses for achieving an optimal process or result
- All variables must be within desirable limits to provide the best possible outcome
- Priorities must be stated by stating the importance of a given variable to achieve a response
- s is the weight or power value set to establish the importance of a given variable to achieve a response closest to the maximum.
- t is the weight to establish how important is for the response Y to be close to the minimum value.
- Ti is the target value for the most desirable response
- D value= 0-1, where 0 states for an undesirable response, and 1 represents an ideal response.

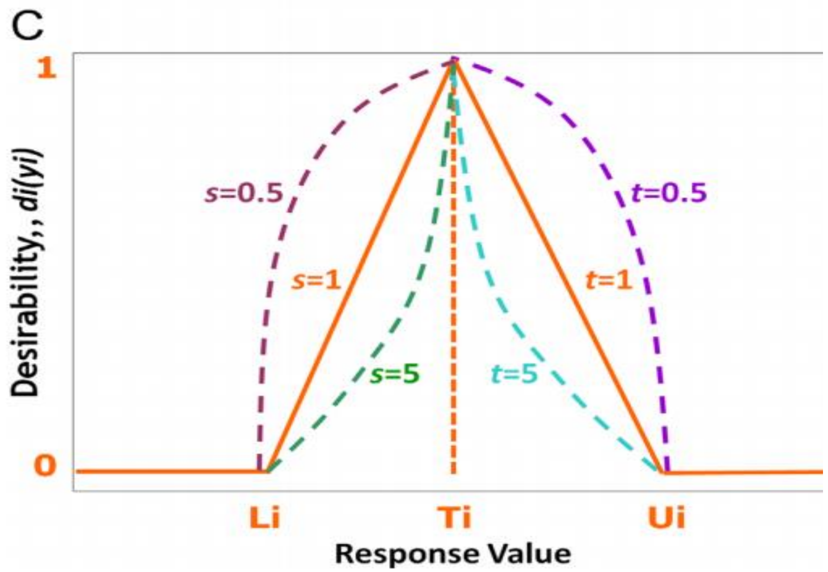
Graphical representation of the desirability functions for the different optimization criteria



Maximized response



Minimized response



Target value (Ti)

- ❑ Ui is the upper acceptable value for the response
- ❑ Li is the lower acceptable value for the response

Conclusions

- DOE helps to reduce experimental work while maximising the potential of the results and its analysis.
- DOE via factorial and optimisation designs is widely used
- DOE allows the simultaneous study of multiple variables while identifying the most important ones
- It is important to become familiar with the statistical concepts behind DOE and the use of statistical software
- As a researcher, DOE could enrich the quality of our work

Session 3
Case study of food waste HTC outputs

Session 2- Creating the DOE for HTC

Selecting the factors:
- Temperature
- Solid load
- Reaction time
- pH
- Catalyst load

Select a DOE based on
- Number of factors
- Amount of sample
- Responses analysis
- Orthogonality/Rotability

Most common DOE for RSM optimization (Candioti et al., 2014)

Design	Type of factors	Factor levels	Number of experiments	Orthoganility	Rotability
Central composite (CCD)	Numerical Categorical	5	$2^k + 2k + Cp$	Yes - No	Yes - No
Box-Behnken (BBD)	Numerical Categorical	3	2k(k-1) + Cp	Yes	Yes
Full factorial design at three level (3-FFD)	Numerical Categorical	3	3^k	Completely ortogonal	No
Doehlert matrix (DMD)	Numerical Categorical	Different for each factor	$K^2+k + Cp$	No	No
D-Optimal	Numerical Categorcal	Different for each model. Irregular experimantal domains	Selected subset of all posible combinations	No	Yes

Session 2-Creating the DOE for HTC

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Create run set, using a appropriate software Interface user-friendly statistical packages:

- Design-Expert
- MiniTab

Input:

1. Design type
2. Factors
3. Levels

Example: run set of a Central composite rotatable design, 3 factors. 8 cubic points, 6 axial points and 6 centre points

Coded values			Actual values		
Temperature (°C)	Reaction time (min)	Moisture content (%)	Temperature (°C)	Reaction time (min)	Moisture content (%)
-1	-1	-1	180	20	75
1	-1	-1	240	20	75
-1	1	-1	180	60	75
1	1	-1	240	60	75
-1	-1	1	180	20	85
1	-1	1	240	20	85
-1	1	1	180	60	85
1	1	1	240	60	85
-α	0	0	159.54	40	80
α	0	0	260.45	40	80
0	-α	0	210	6.36	80
0	α	0	210	73.63	80
0	0	-α	210	40	71.59
0	0	α	210	40	88.40
0	0	0	210	40	80
0	0	0	210	40	80
0	0	0	210	40	80
0	0	0	210	40	80
0	0	0	210	40	80
0	0	0	210	40	80

Session 2 - Example

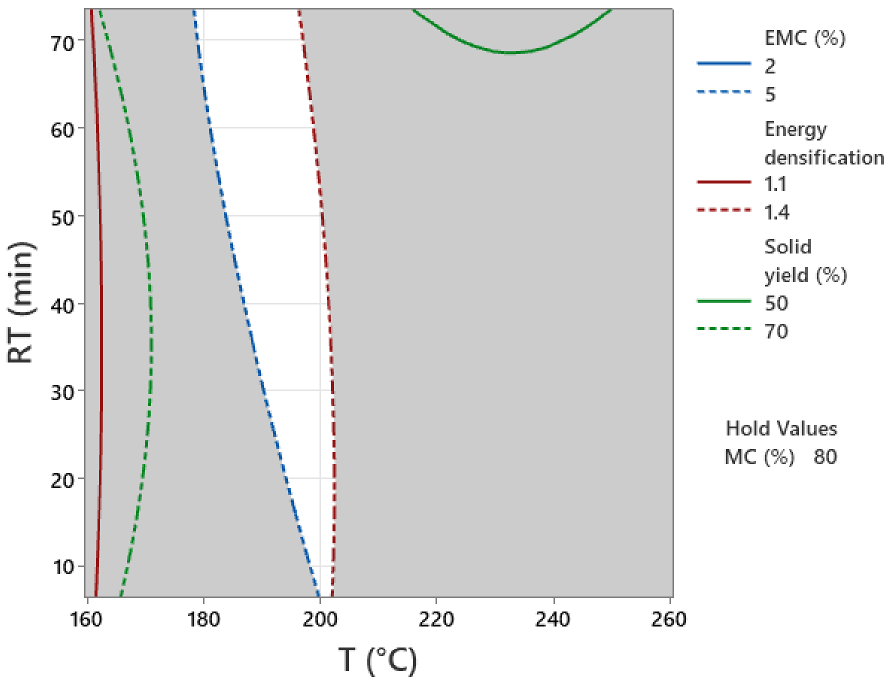
Actual values		
Temperature (°C)	Reaction time (min)	Moisture content (%)
180	20	75
240	20	75
180	60	75
240	60	75
180	20	85
240	20	85
180	60	85
240	60	85
159.54	40	80
260.45	40	80
210	6.36	80
210	73.63	80
210	40	71.59
210	40	88.40
210	40	80
210	40	80
210	40	80
210	40	80
210	40	80
210	40	80

Optimize for :

- Solid yield (%)
- Energy densification
- Equilibrium moisture content

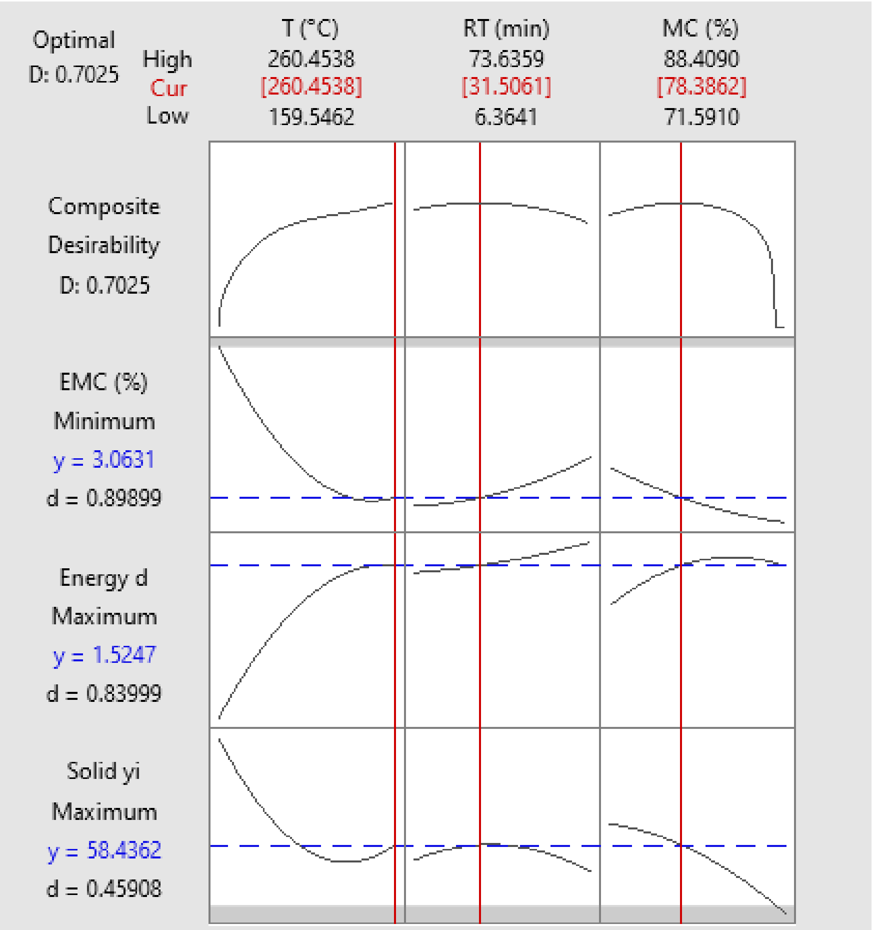
Solid yield (%)	Energy densification	EMC (%)
66.18	1.19	8.19
55.29	1.40	3.83
63.27	1.24	4.75
57.09	1.49	3.82
47.90	1.31	4.45
51.81	1.52	2.44
53.11	1.30	4.27
49.24	1.52	2.71
88.06	1.00	8.57
50.71	1.62	2.71
57.72	1.45	3.77
51.67	1.47	4.56
63.84	1.32	5.24
47.30	1.42	2.94
57.57	1.45	2.91
53.21	1.48	3.99
57.32	1.49	2.98
57.14	1.43	4.46
53.92	1.44	3.90
55.61	1.36	3.10

Session 2 - Example



- Generates an optimized area for hydrochar production
- Useful for working with ranges of the response
  - Ranges are shortened based on the product of interest
- Only two process factors are evaluated at a time
  - Most useful for working with 2 factors

Session 2 - Example



Criteria:

Solid yield: maximize

Energy densification: maximize

Equilibrium moisture content: minimize

- All factors are considered in the optmization calculations
- The hierarchy of the responese is adjustable
- Determines an optimized point instead of an area

Session 2 - References

Candioti, L.V., De Zan, M.M., Cámara, M.S. and Goicoechea, H.C., 2014. Experimental design and multiple response optimization. Using the desirability function in analytical methods development. *Talanta*, *124*, pp.123-138.

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References

Candioti et al. (2014) Review Experimental design and multiple response optimization. Using the desirability function in analytical methods development. Talanta 124; 123-138.

Mikko (2017) Experimental design and response surface methodology in energy applications: A tutorial review. Energy Conversion and management 151; 630-640.

Montgomery (2013) Design and analysis of experiments. Eight Edition.

References

Akarsu, K., Duman, G., Yilmazer, A., Keskin, T., Azbar, N., Yanik, J., 2019. Sustainable valorization of food wastes into solid fuel by hydrothermal carbonization. Bioresour. Technol. 292, 121959. https://doi.org/10.1016/j.biortech.2019.121959

Álvarez-Murillo, A., Román, S., Ledesma, B., Sabio, E., 2015. Study of variables in energy densification of olive stone by hydrothermal carbonization. J. Anal. Appl. Pyrolysis 113, 307–314. https://doi.org/10.1016/j.jaap.2015.01.031

Bhakta Sharma, H., Panigrahi, S., Dubey, B.K., 2021. Food waste hydrothermal carbonization: Study on the effects of reaction severities, pelletization and framework development using approaches of the circular economy. Bioresour. Technol. 333, 125187. https://doi.org/10.1016/j.biortech.2021.125187

Chen, X., Lin, Q., He, R., Zhao, X., Li, G., 2017. Hydrochar production from watermelon peel by hydrothermal carbonization. Bioresour. Technol. 241, 236–243. https://doi.org/10.1016/j.biortech.2017.04.012

Danso-Boateng, E., Shama, G., Wheatley, A.D., Martin, S.J., Holdich, R.G., 2015. Hydrothermal carbonisation of sewage sludge: Effect of process conditions on product characteristics and methane production. Bioresour. Technol. 177, 318–327. https://doi.org/10.1016/j.biortech.2014.11.096

Feng, Y., Sun, H., Han, L., Xue, L., Chen, Y., Yang, L., Xing, B., 2019. Fabrication of hydrochar based on food waste (FWHTC) and its application in aqueous solution rare earth ions adsorptive removal: Process, mechanisms and disposal methodology. J. Clean. Prod. 212, 1423–1433. https://doi.org/10.1016/j.jclepro.2018.12.094

Gupta, D., Mahajani, S.M., Garg, A., 2020. Investigation on hydrochar and macromolecules recovery opportunities from food waste after hydrothermal carbonization. Sci. Total Environ. 749, 142294. https://doi.org/10.1016/j.scitotenv.2020.142294

Kannan, S., Gariepy, Y., Vijaya Raghavan, G.S., 2018. Optimization of the conventional hydrothermal carbonization to produce hydrochar from fish waste. Biomass Convers. Biorefinery 8, 563–576. https://doi.org/10.1007/s13399-018-0323-9

Li, Y., Liu, H., Xiao, K., Jin, M., Xiao, H., Yao, H., 2020. Combustion and Pyrolysis Characteristics of Hydrochar Prepared by Hydrothermal Carbonization of Typical Food Waste: Influence of Carbohydrates, Proteins, and Lipids. Energy and Fuels 34, 430–439. https://doi.org/10.1021/acs.energyfuels.9b02940

Lin, Y., Ge, Y., Xiao, H., He, Q., Wang, W., Chen, B., 2020. Investigation of hydrothermal co-carbonization of waste textile with waste wood, waste paper and waste food from typical municipal solid wastes. Energy 210. https://doi.org/10.1016/j.energy.2020.118606

Lucian, M., Volpe, M., Merzari, F., Wüst, D., Kruse, A., Andreottola, G., Fiori, L., 2020. Hydrothermal carbonization coupled with anaerobic digestion for the valorization of the organic fraction of municipal solid waste. Bioresour. Technol. 314, 123734. https://doi.org/10.1016/j.biortech.2020.123734

Mahmood, R., Parshetti, G.K., Balasubramanian, R., 2016. Energy, exergy and techno-economic analyses of hydrothermal oxidation of food waste to produce hydro-char and bio-oil. Energy 102, 187–198. https://doi.org/10.1016/j.energy.2016.02.042

Mazumder, S., Saha, P., McGaughy, K., Saba, A., Reza, M.T., 2020. Technoeconomic analysis of co-hydrothermal carbonization of coal waste and food waste. Biomass Convers. Biorefinery. https://doi.org/10.1007/s13399-020-00817-8

McGaughy, K., Toufiq Reza, M., 2018. Hydrothermal carbonization of food waste: simplified process simulation model based on experimental results. Biomass Convers. Biorefinery 8, 283–292. https://doi.org/10.1007/s13399-017-0276-4

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References

Picone, A., Volpe, M., Giustra, M.G., Bella, G. Di, Messineo, A., 2021. Hydrothermal carbonization of lemon peel waste: Preliminary results on the effects of temperature during process water recirculation. Appl. Syst. Innov. 4. https://doi.org/10.3390/asi4010019

Sabio, E., Álvarez-Murillo, A., Román, S., Ledesma, B., 2016. Conversion of tomato-peel waste into solid fuel by hydrothermal carbonization: Influence of the processing variables. Waste Manag. 47, 122–132. https://doi.org/10.1016/j.wasman.2015.04.016

Saqib, N.U., Baroutian, S., Sarmah, A.K., 2018. Physicochemical, structural and combustion characterization of food waste hydrochar obtained by hydrothermal carbonization. Bioresour. Technol. 266, 357–363. https://doi.org/10.1016/j.biortech.2018.06.112

Sharma, H.B., Dubey, B.K., 2020. Co-hydrothermal carbonization of food waste with yard waste for solid biofuel production: Hydrochar characterization and its pelletization. Waste Manag. 118, 521–533. https://doi.org/10.1016/j.wasman.2020.09.009

Solomon, P.R., 1981. Relation between coal aromatic carbon concentration and proximate analysis fixed carbon. Fuel 60, 3–6. https://doi.org/10.1016/0016-2361(81)90023-5

Stutzenstein, P., Bacher, M., Rosenau, T., Pfeifer, C., 2018. Optimization of Nutrient and Carbon Recovery from Anaerobic Digestate via Hydrothermal Carbonization and Investigation of the Influence of the Process Parameters. Waste and Biomass Valorization 9, 1303–1318. https://doi.org/10.1007/s12649-017-9902-4

Toptas Tag, A., Duman, G., Yanik, J., 2018. Influences of feedstock type and process variables on hydrochar properties. Bioresour. Technol. 250, 337–344. https://doi.org/10.1016/j.biortech.2017.11.058

Tradler, S.B., Mayr, S., Himmelsbach, M., Priewasser, R., Baumgartner, W., Stadler, A.T., 2018. Hydrothermal carbonization as an all-inclusive process for food-waste conversion. Bioresour. Technol. Reports 2, 77–83. https://doi.org/10.1016/j.biteb.2018.04.009

Volpe, M., Fiori, L., 2017. From olive waste to solid biofuel through hydrothermal carbonisation: The role of temperature and solid load on secondary char formation and hydrochar energy properties. J. Anal. Appl. Pyrolysis 124, 63–72. https://doi.org/10.1016/j.jaap.2017.02.022

Wang, T., Zhai, Y., Zhu, Y., Gan, X., Zheng, L., Peng, C., Wang, B., Li, C., Zeng, G., 2018a. Evaluation of the clean characteristics and combustion behavior of hydrochar derived from food waste towards solid biofuel production. Bioresour. Technol. 266, 275–283. https://doi.org/10.1016/j.biortech.2018.06.093

Wang, T., Zhai, Y., Zhu, Y., Peng, C., Xu, B., Wang, T., Li, C., Zeng, G., 2018b. Influence of temperature on nitrogen fate during hydrothermal carbonization of food waste. Bioresour. Technol. 247, 182–189. https://doi.org/10.1016/j.biortech.2017.09.076

Wilk, M., Śliz, M., Gajek, M., 2021. The effects of hydrothermal carbonization operating parameters on high-value hydrochar derived from beet pulp. Renew. Energy 177, 216–228. https://doi.org/10.1016/j.renene.2021.05.112