## Approaches to Treatment Optimization of Humics in Wastewater: 'DOE' Vs 'OFAT'



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#### Fresh-Cut Agricultural Produce

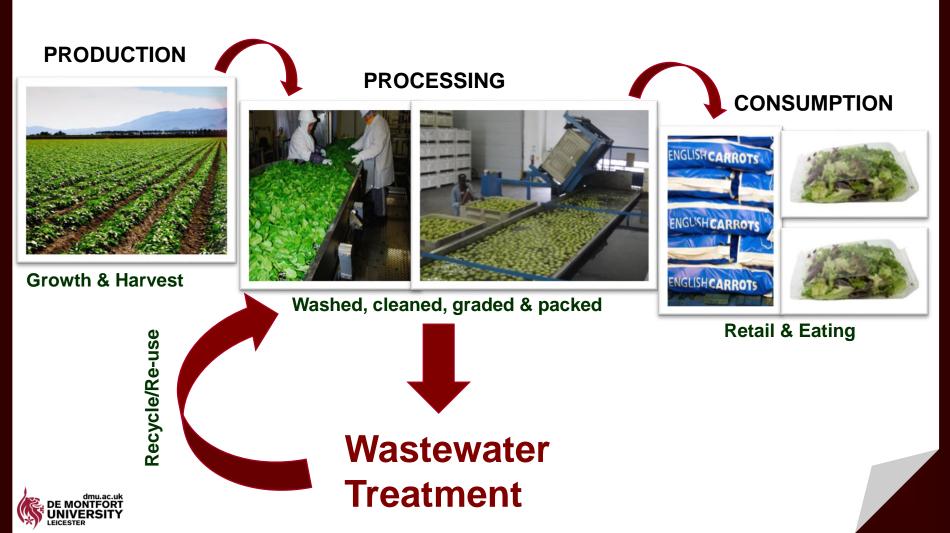
 Any fruit or vegetable that undergoes minimal or no processing after harvest







#### Fresh-Cut Agricultural Industry



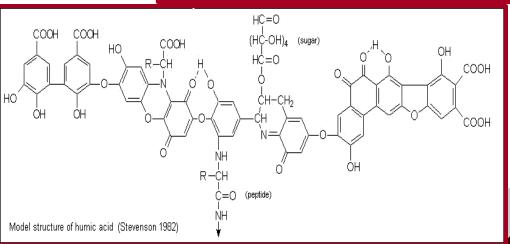
#### Fresh-Cut Produce Wastewater

- High amount of organic load
  - Field soil
  - Plant debris
  - Soil particles
  - Salt
  - Pesticide residues



Humic substances





#### What the Legislation Says?

- Reuse and recycling of wastewater is supported by European Union (Council Directive, 1991; 1998; EU, 1996)
- Use of alternative water qualities is justified
  - Reuse must not compromise sanitation
  - Reuse must not cause adulteration of the product



- Wastewater reuse in food industries for washing purposes only
  - Wastewater must be treated by an advanced treatment system
  - Product, facilities, equipment, and utensils must undergo a separate final rinse with fresh portable water (USDA, 1999)



#### Advanced Oxidation

- Advanced Chemical Oxidation
  - Heterogeneous Catalyst







http://www.tucsonaz.gov/water/aop

Lab scale



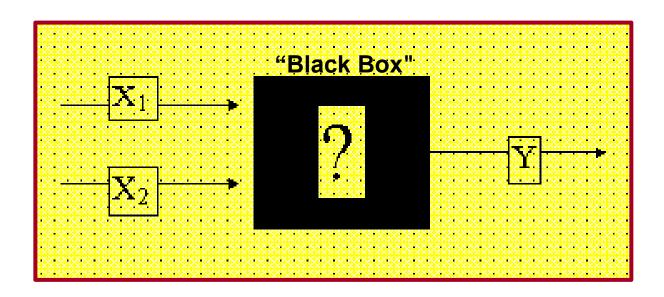
Pilot scale



**Commercial scale** 



#### **Experimental Optimization**



- Wastewater (humics)
- Catalyst
- H<sub>2</sub>O<sub>2</sub>
- pH

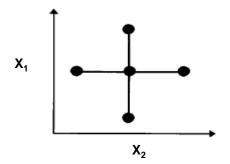
Chemical process (Catalysis)

**Treated wastewater** 



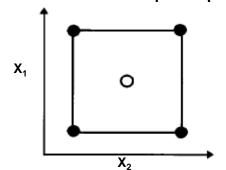
## Optimization Approaches

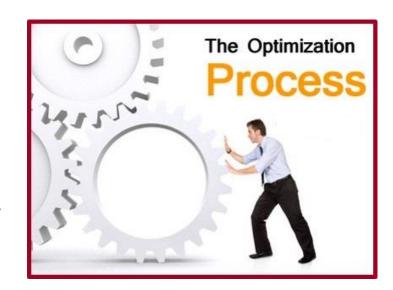
- 'One Factor at a time' (OFAT)
  - -Main-Effects Model:  $Y_1 = X_1 + X_2$



Design of experiments (DOE)

-Statistically vary factors simultaneously Interactions Model:  $Y_1 = X_1 + X_2 + X_1*X_2$ 







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#### What We Did

Compared a statistical design of experiment (DOE) and one factor at a time (OFAT) approach in the optimization of humics contaminated water





### The Questions



Will the DOE and OFAT approach provide the same optimum treatment condition for humics?



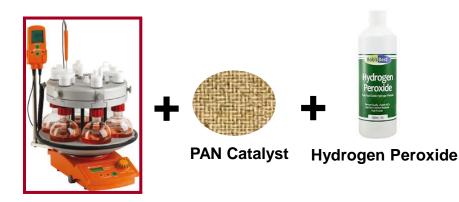
Which approach is better?

- Treatment efficiency
- Faster
- Cost savings





#### How We Did It







- Bench scale experiments
- Simulated Humic acid contaminated water (25mg/L)
- Key parameters
  - pH
  - H<sub>2</sub>O<sub>2</sub> concentration
  - Mass of Catalyst
- UV/Vis Humic acid
  - Aromaticity @254 nm
  - Colour @400 nm

•HPLC-UV - H<sub>2</sub>O<sub>2</sub>



#### How We Did It

The Box–Behnken design

JMP Pro 11 software



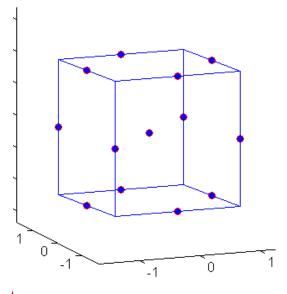
Vay Daramatara	Levels		
Key Parameters	Low (-1)	Center (0)	High (+1)
Mass of Catalyst (g)	3	5	7
$H_2O_2$ (mg/L)	50	550	1000
рН	3	5	7
Responses	Goal		
% Degradation of Humic acid @ 254nm	Maximize		
% Degradation of Humic acid @ 400nm	Maximize		



#### How We Did It

#### The Box–Behnken design

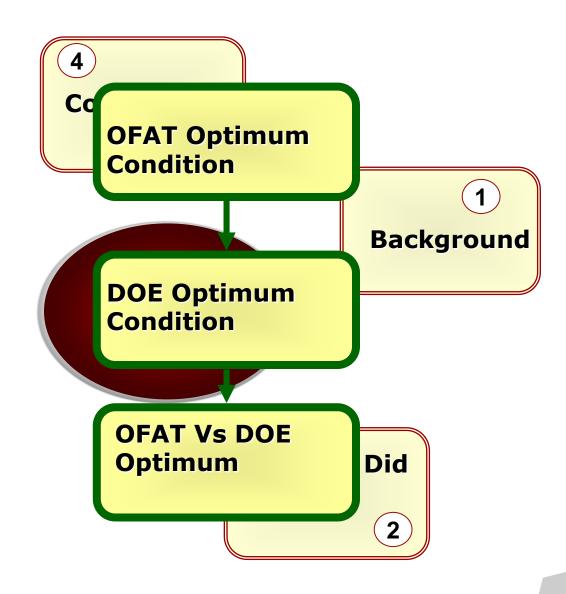
- 15 experiments
- 3 replicated center points



	Mode	Key Parameters			
Run		Catalyst (g)	H <sub>2</sub> O <sub>2</sub> (mg/L)	рН	
1	+ 0 +	7	550	3	
2	0 + -	5	1000	3	
3	0	5	100	3	
4	- 0 -	3	550	3	
5	0	3	100	5	
6	++0	7	1000	5	
7	- + 0	3	1000	5	
8	+ - 0	7	100	5	
9	0 + +	5	1000	7	
10	0 - +	5	100	7	
11	+ 0 +	7	550	7	
12	- 0 +	3	550	7	
13	0 0 0	5	550	5	
14	0 0 0	5	550	5	
15	0 0 0	5	550	5	







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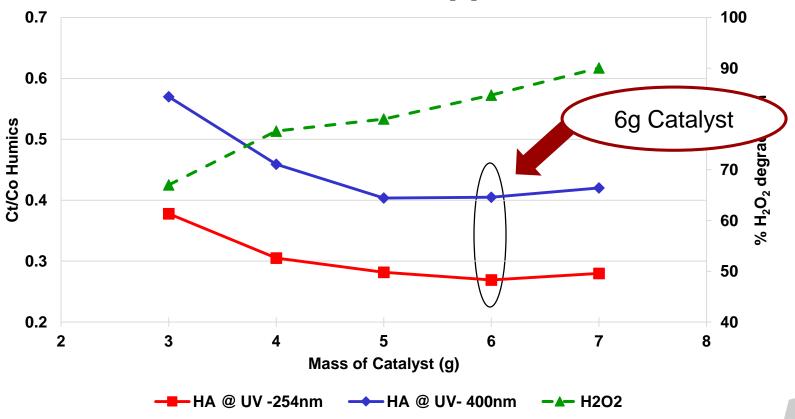
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#### OFAT Optimum Catalyst

#### EFFECT OF CATALYST ON HUMICS DEGRADATION

(Vol. 100mg/L; 25mg/L HA; 500mg/L H<sub>2</sub>O<sub>2</sub>; pH 5.3; Temp 23°C)

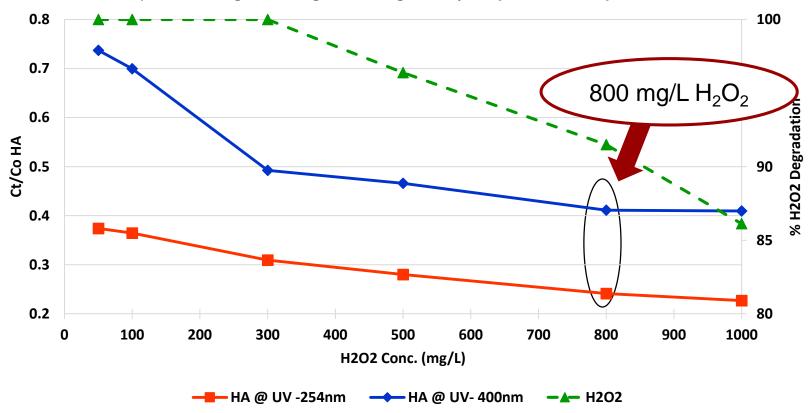




#### OFAT Optimum H<sub>2</sub>O<sub>2</sub>

#### EFFECT OF H<sub>2</sub>O<sub>2</sub> ON HUMICS DEGRADATION

(Vol. 100mg/L; 25mg/L HA; 6g catalyst; pH 5.3; Temp 23°C)

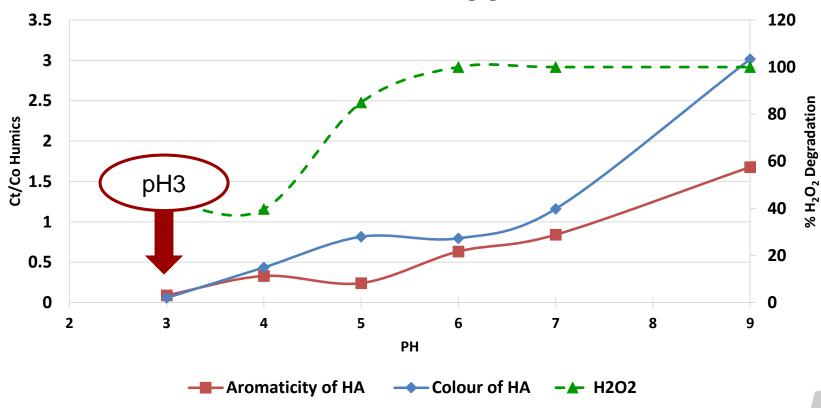




#### OFAT Optimum pH

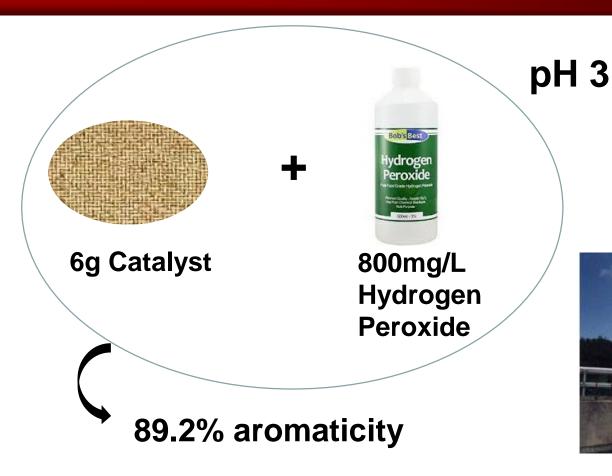
#### **EFFECT OF PH ON HUMICS DEGRADATION**

(Vol. 100ml; 25mg/L HA; 800mg/L H<sub>2</sub>O<sub>2</sub>; 6g Catalyst; 23°C)





## OFAT Optimum

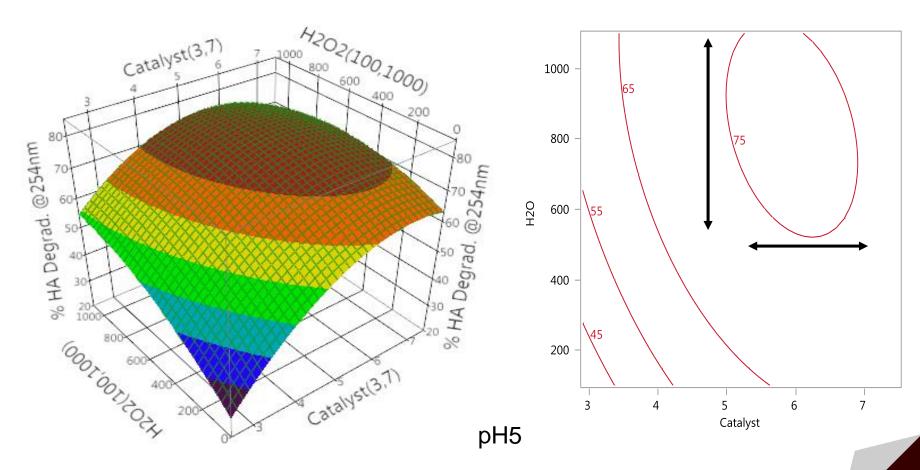






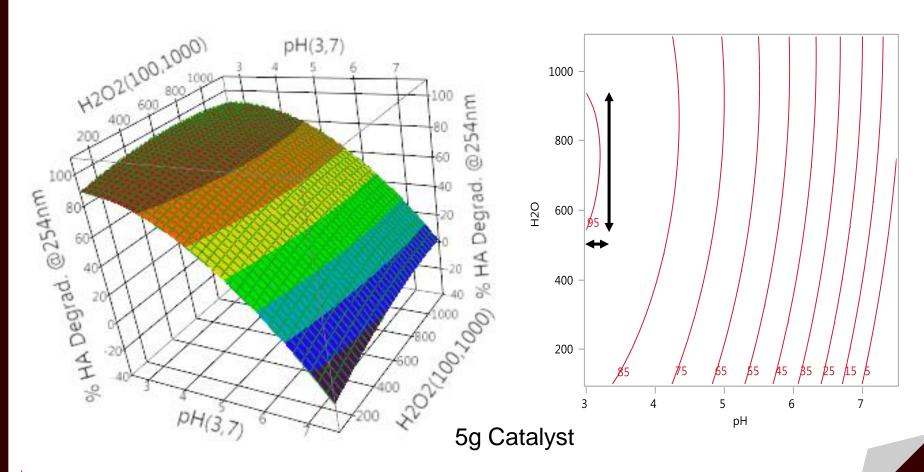
94.2% colour

#### DOE Optimum Catalyst



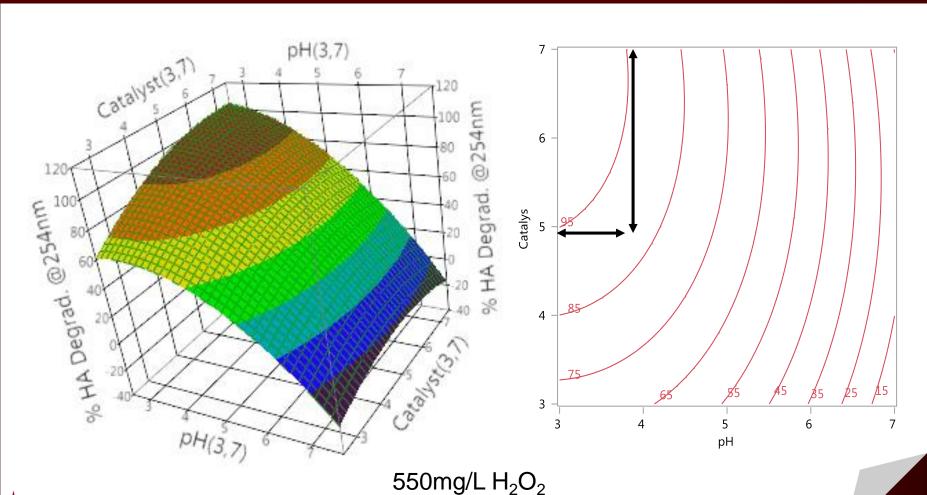


## DOE Optimum H<sub>2</sub>O<sub>2</sub>



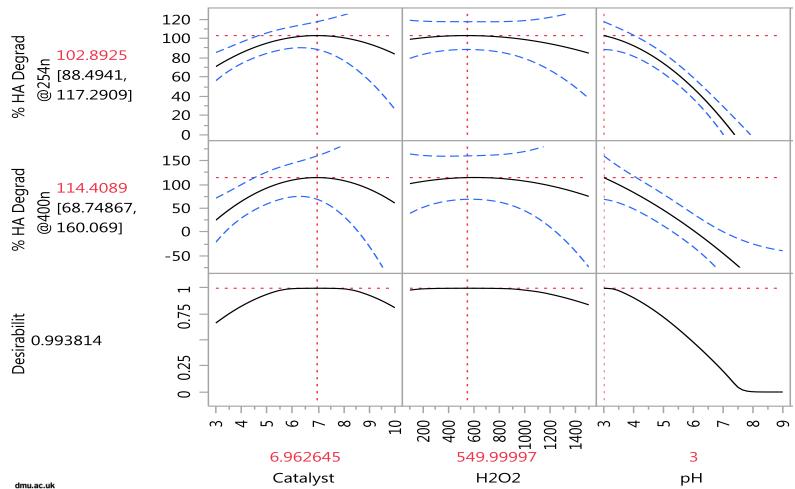


#### DOE Optimum pH



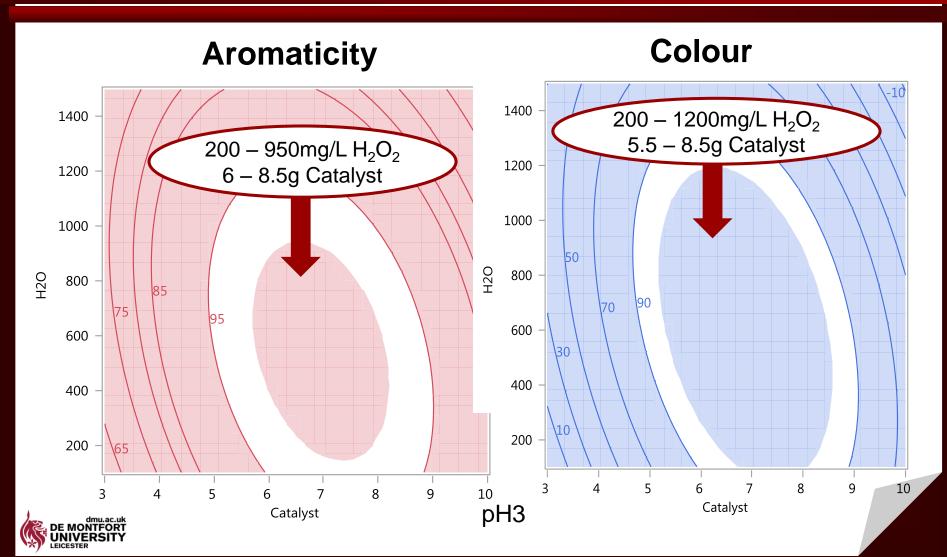


## DOE Optimum



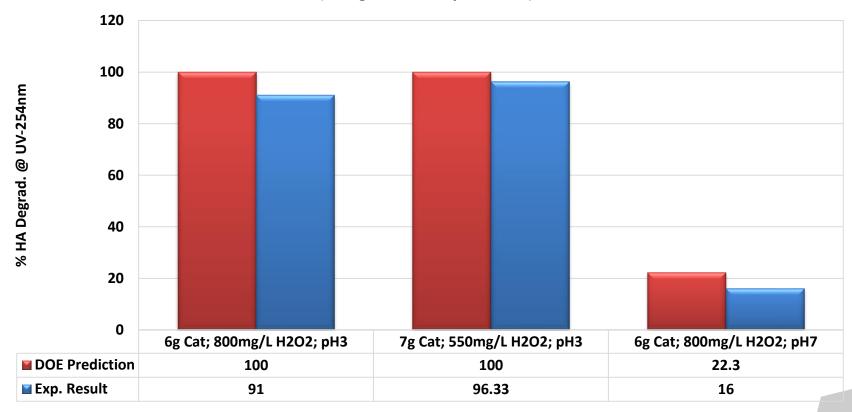


## DOE Optimum



#### Validation of DOE

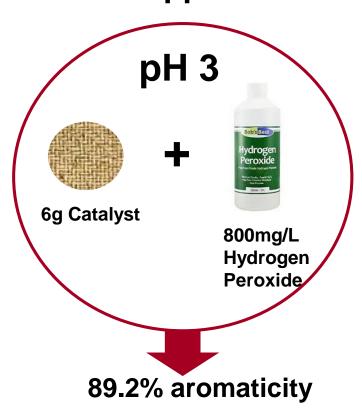
#### Validation of DOE Conditions of Humic Acid Degradation (25mg/L HA; Temp 22±1 0C)





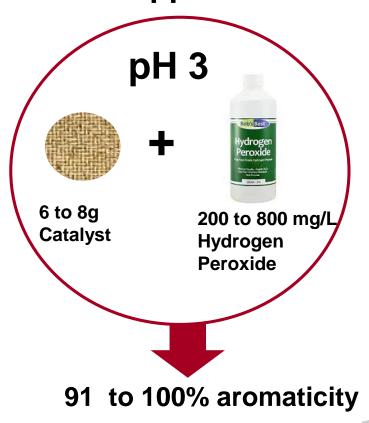
#### OFAT Vs DOE

#### **OFAT Approach**



94.2% colour

#### **DOE Approach**



100% colour

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Will the OFAT and DOE approaches provide the same optimum treatment condition for wastewater?



OFAT – 800mg/L  $H_2O_2$ ; 6g Catalyst; pH 3 DOE – Range of conditions: 200 to 800mg/L  $H_2O_2$ ; 6 to 8.5g Catalyst; pH 3 to 3.5





Which approach is better?

- The DOE approach to optimization was more robust
  - More information on the design space
  - Cost savings: ≈ 4 folds (75%) savings on hydrogen peroxide



# Thank You

