

A Novel Method to Detoxify Steam Exploded Biomass and Produce a Substrate for Biorefinery

Francesco Zimbardi *, Egidio Viola, G. Arcieri, Vito Valerio, and Massimo Carnevale

Detoxification with the Pilot scale Vibro-fluidized bed system: detailed data by DOE

Table S1. Experimental responses at different conditions

| Exp # | Ur,% | t,min | DM,% | Acetic acid, % | Furfural, ppm | 5-HMF, ppm | Total inhibitors, % | Enzymatic hydrolysis yield, % |
|-------|------|-------|------|-------------------|------------------|---------------|------------------------|----------------------------------|
| 1 | 80 | 50 | 83.5 | 0.079 | 29 | 537 | 0.136 | 80.6 |
| 2 | 60 | 10 | 81.5 | 0.379 | 60 | 433 | 0.428 | 88.2 |
| 3 | 60 | 50 | 84.1 | 0.166 | 36 | 366 | 0.206 | 83.6 |
| 4 | 40 | 30 | 94.7 | 0.155 | 43 | 391 | 0.198 | 81.1 |
| 5 | 80 | 30 | 87.5 | 0.049 | 32 | 433 | 0.096 | 85.7 |
| 6 | 80 | 10 | 70.8 | 0.323 | 42 | 540 | 0.381 | 85.7 |
| 7 | 40 | 10 | 92.6 | 0.35 | 69 | 592 | 0.416 | 85.6 |
| 8 | 60 | 30 | 93.2 | 0.11 | 36 | 576 | 0.171 | 88.8 |
| 9 | 40 | 50 | 94.9 | 0.155 | 64 | 598 | 0.375 | 86.2 |
| 10 | 60 | 30 | 95.5 | 0.11 | 35 | 591 | 0.172 | 84.3 |

Design-Expert® Software
Factor Coding: Actual
Acetic acid (%)



X1 = A: Ur
X2 = B: t

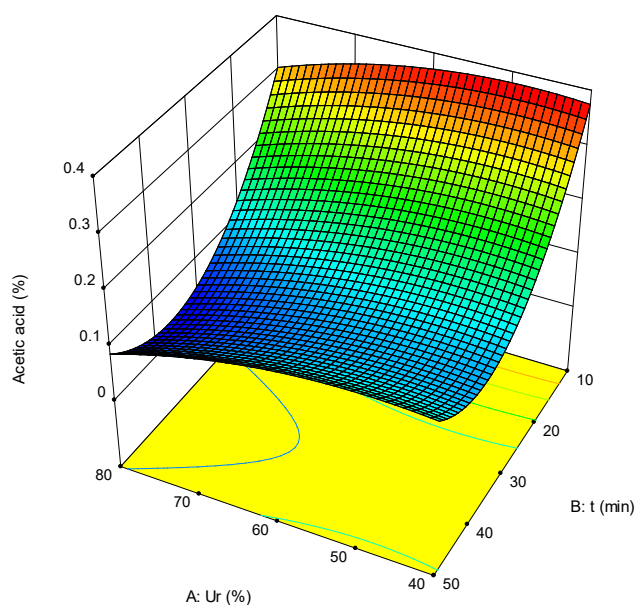


Figure S1. Acetic acid in the detoxified pulp with the pilot vibro-fluidized bed at different treatment conditions.

Design-Expert® Software
Factor Coding: Actual
Furfural (ppm)



X1 = A: Ur
X2 = B: t

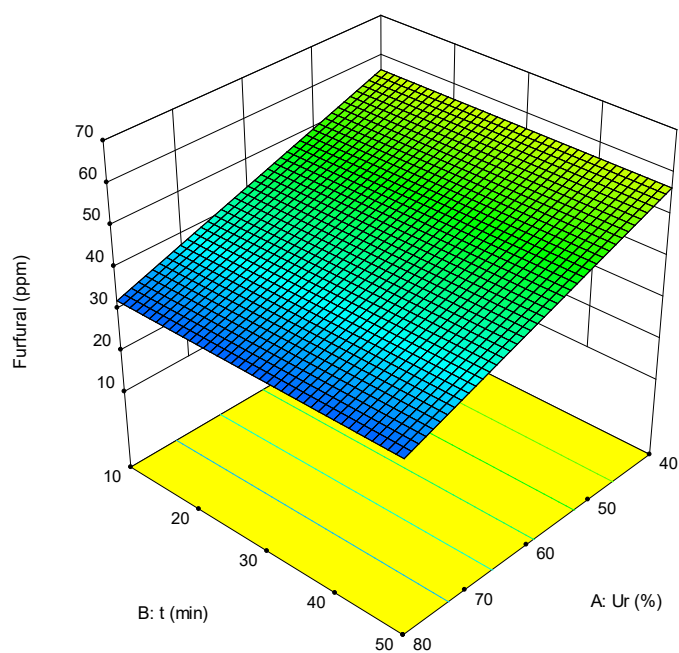


Figure S2. Furfural in the detoxified pulp with the pilot vibro-fluidized bed at different treatment conditions.

Design-Expert® Software
Factor Coding: Actual
5-HMF (ppm)



X1 = A: Ur
X2 = B: t

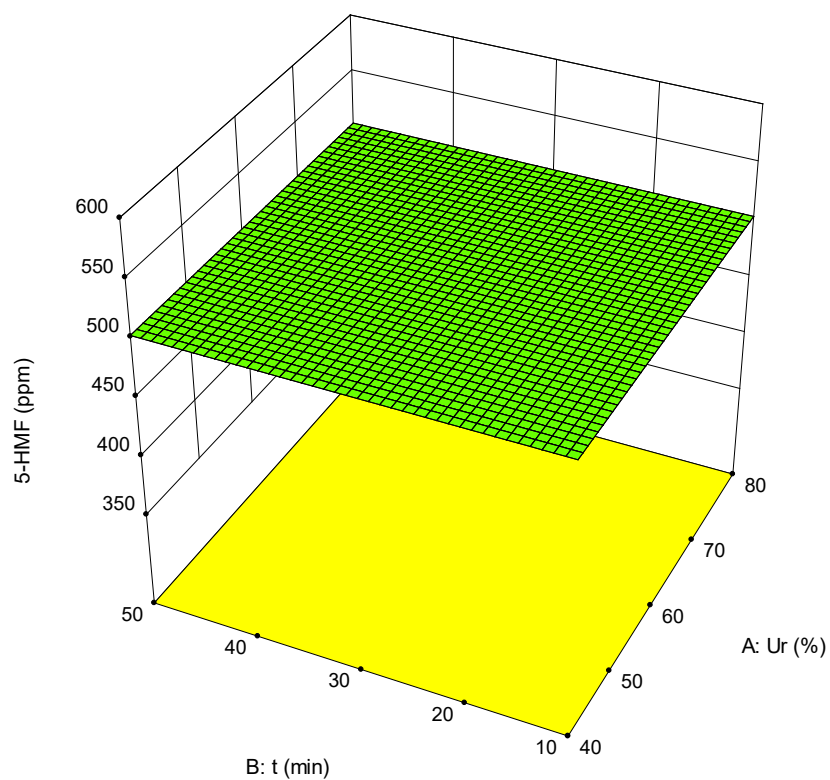
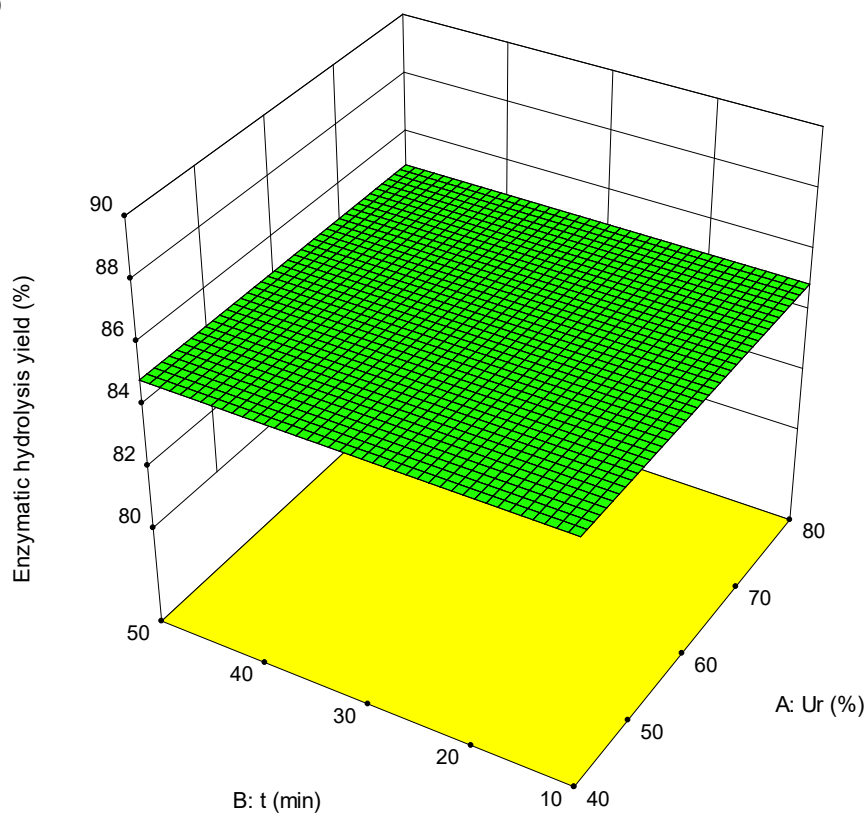


Figure S3. 5-HMF in the detoxified pulp with the pilot vibro-fluidized bed at different treatment conditions.

Design-Expert® Software
 Factor Coding: Actual
 Enzymatic hydrolysis yield (%)



X1 = A: Ur
 X2 = B: t



Design-Expert® Software
 Factor Coding: Actual
 Enzymatic hydrolysis yield (%)
 --- 95% CI Bands

Actual Factors
 A: Ur = 60
 B: t = 30

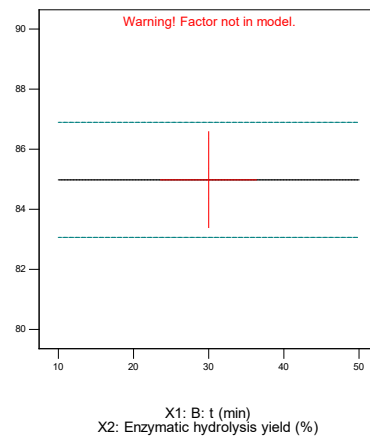
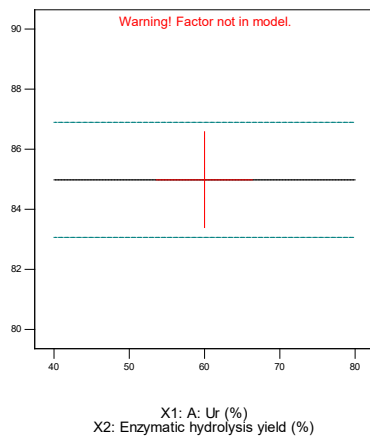


Figure S4. Predicted EH yield (%) on the detoxified pulp at different treatment conditions..

Table S2. ANOVA for DM

| ANOVA for Response Surface Reduced Quadratic model | | | | | | |
|--|----------------|----|-------------|---------|------------------|-----------------|
| Analysis of variance table [Partial sum of squares - Type III] | | | | | | |
| Source | Sum of Squares | df | Mean Square | F Value | p-value Prob > F | |
| Model | 431.77 | 2 | 215.88 | 10.90 | 0.0071 | significant |
| A-Ur | 272.03 | 1 | 272.03 | 13.73 | 0.0076 | |
| B² | 159.74 | 1 | 159.74 | 8.06 | 0.0251 | |
| Residual | 138.69 | 7 | 19.81 | | | |
| Lack of Fit | 136.05 | 6 | 22.67 | 8.57 | 0.2557 | not significant |
| Pure Error | 2.64 | 1 | 2.64 | | | |
| Cor Total | 570.46 | 9 | | | | |

Table S3. Coefficients for DM

| Coefficient | | Standard | | 95% CI | | VIF |
|----------------------|----------|----------|-------|--------|-------|------|
| Factor | Estimate | df | Error | Low | High | |
| Intercept | 92.72 | 1 | 2.23 | 87.46 | 97.99 | |
| A-Ur | -6.73 | 1 | 1.82 | -11.03 | -2.44 | 1.00 |
| B² | -8.16 | 1 | 2.87 | -14.95 | -1.36 | 1.00 |

Table S4. ANOVA for Inhibitors

| ANOVA for Response Surface Reduced Quadratic model | | | | | | |
|--|----------------|----|-------------|---------|------------------|-------------|
| Analysis of variance table [Partial sum of squares - Type III] | | | | | | |
| Source | Sum of Squares | df | Mean Square | F Value | p-value Prob > F | |
| Model | 0.14 | 4 | 0.035 | 55.02 | 0.0003 | significant |
| A-Ur | 0.024 | 1 | 0.024 | 36.56 | 0.0018 | |
| B-t | 0.043 | 1 | 0.043 | 66.73 | 0.0004 | |
| AB | 0.010 | 1 | 0.010 | 16.14 | 0.0101 | |
| B² | 0.065 | 1 | 0.065 | 100.66 | 0.0002 | |
| Residual | 3.223E-003 | 5 | 6.445E-004 | | | |
| Lack of Fit | 3.222E-003 | 4 | 8.056E-004 | 1611.13 | 0.0187 | significant |
| Pure Error | 5.000E-007 | 1 | 5.000E-007 | | | |
| Cor Total | 0.15 | 9 | | | | |

Table S5. Coefficients for Inhibitors

| Coefficient | | Standard | | 95% CI | | VIF |
|----------------------|----------|----------|-------|--------|--------|------|
| Factor | Estimate | df | Error | Low | High | |
| Intercept | 0.16 | 1 | 0.013 | 0.13 | 0.19 | |
| A-Ur | -0.063 | 1 | 0.010 | -0.089 | -0.036 | 1.00 |
| B-t | -0.085 | 1 | 0.010 | -0.11 | -0.058 | 1.00 |
| AB | -0.051 | 1 | 0.013 | -0.084 | -0.018 | 1.00 |
| B² | 0.16 | 1 | 0.016 | 0.12 | 0.21 | 1.00 |

Table S6. ANOVA for Acetic acid

| ANOVA for Response Surface Reduced Quadratic model | | | | | | |
|--|----------------|----|-------------|---------|------------------|-------------|
| Analysis of variance table [Partial sum of squares - Type III] | | | | | | |
| Source | Sum of Squares | df | Mean Square | F Value | p-value Prob > F | |
| Model | 0.12 | 4 | 0.031 | 64.37 | 0.0002 | significant |
| A-Ur | 7.280E-003 | 1 | 7.280E-003 | 15.05 | 0.0116 | |
| B-t | 0.071 | 1 | 0.071 | 146.46 | < 0.0001 | |
| A² | 2.040E-003 | 1 | 2.040E-003 | 4.22 | 0.0952 | |
| B² | 0.046 | 1 | 0.046 | 95.80 | 0.0002 | |
| Residual | 2.419E-003 | 5 | 4.837E-004 | | | |
| Lack of Fit | 2.419E-003 | 4 | 6.047E-004 | | | |
| Pure Error | 0.000 | 1 | 0.000 | | | |
| Cor Total | 0.13 | 9 | | | | |

Table S7. Coefficients for Acetic acid

| Coefficient | | Standard | | 95% CI | | VIF |
|----------------------|----------|----------|------------|--------|------------|------|
| Factor | Estimate | df | Error | Low | High | |
| Intercept | 0.12 | 1 | 0.013 | 0.087 | 0.15 | |
| A-Ur | -0.035 | 1 | 8.979E-003 | -0.058 | -0.012 | 1.00 |
| B-t | -0.11 | 1 | 8.979E-003 | -0.13 | -0.086 | 1.00 |
| A² | -0.030 | 1 | 0.014 | -0.067 | 7.441E-003 | 1.03 |
| B² | 0.14 | 1 | 0.014 | 0.10 | 0.18 | 1.03 |

Table S8. ANOVA for furfural

| ANOVA for Response Surface Reduced Linear model | | | | | | |
|--|----------------|----|-------------|---------|------------------|-------------|
| Analysis of variance table [Partial sum of squares - Type III] | | | | | | |
| Source | Sum of Squares | df | Mean Square | F Value | p-value Prob > F | |
| Model | 888.17 | 1 | 888.17 | 7.31 | 0.0269 | significant |
| A-Ur | 888.17 | 1 | 888.17 | 7.31 | 0.0269 | |
| Residual | 972.23 | 8 | 121.53 | | | |
| Lack of Fit | 971.73 | 7 | 138.82 | 277.64 | 0.0462 | significant |
| Pure Error | 0.50 | 1 | 0.50 | | | |
| Cor Total | 1860.40 | 9 | | | | |

Table S9. Coefficients for Furfural

| Coefficient | | Standard | | 95% CI | | VIF |
|------------------|----------|----------|-------|--------|-------|------|
| Factor | Estimate | df | Error | Low | High | |
| Intercept | 44.60 | 1 | 3.49 | 36.56 | 52.64 | |
| A-Ur | -12.17 | 1 | 4.50 | -22.54 | -1.79 | 1.00 |

Table S10. ANOVA for 5-HMF

| ANOVA for Response Surface Mean model | | | | | | |
|--|----------------|----|-------------|---------|---------|-----------------|
| Analysis of variance table [Partial sum of squares - Type III] | | | | | | |
| Source | Sum of Squares | df | Mean Square | F Value | p-value | |
| Model | 0.000 | 0 | | | | |
| Residual | 73584.10 | 9 | 8176.01 | | | |
| Lack of Fit | 73471.60 | 8 | 9183.95 | 81.64 | 0.0854 | not significant |
| Pure Error | 112.50 | 1 | 112.50 | | | |
| Cor Total | 73584.10 | 9 | | | | |

Table S11. Coefficients for 5-HMF

| | Coefficient | | Standard | 95% CI | |
|------------------|-------------|----|----------|--------|--------|
| Factor | Estimate | df | Error | Low | High |
| Intercept | 505.70 | 1 | 28.59 | 441.02 | 570.38 |

Table S12. ANOVA for EH

| ANOVA for Response Surface Mean model | | | | | | |
|--|----------------|----|-------------|---------|---------|-----------------|
| Analysis of variance table [Partial sum of squares - Type III] | | | | | | |
| Source | Sum of Squares | df | Mean Square | F Value | p-value | |
| Model | 0.000 | 0 | | | | |
| Residual | 64.48 | 9 | 7.16 | | | |
| Lack of Fit | 54.35 | 8 | 6.79 | 0.67 | 0.7431 | not significant |
| Pure Error | 10.13 | 1 | 10.13 | | | |
| Cor Total | 64.48 | 9 | | | | |

Table S13. Coefficients for EH

| | Coefficient | | Standard | 95% CI | |
|------------------|-------------|----|----------|--------|-------|
| Factor | Estimate | df | Error | Low | High |
| Intercept | 84.98 | 1 | 0.85 | 83.07 | 86.89 |

Table S14. Coefficients table

| Response | Intercept | A | B | AB | A^2 | B^2 |
|-----------------------------------|-----------|------------|----------------|----------------|------------|----------|
| DM | 92.725 | -6.73333 | | | | -8.15833 |
| p= | | 0.0076 | | | | 0.0251 |
| Inhibitors | 0.15925 | -0.0626667 | -0.0846667 | -0.051 | | 0.164417 |
| p= | | 0.0018 | 0.0004 | 0.0101 | | 0.0002 |
| Acetic acid | 0.120786 | -0.0348333 | -0.108667 | | -0.0295714 | 0.140929 |
| p= | | 0.0116 | < 0.0001 | | 0.0952 | 0.0002 |
| Furfural | 44.6 | -12.1667 | | | | |
| p= | | 0.0269 | | | | |
| 5-HMF | 505.7 | | | | | |
| p= | | | | | | |
| Enzymatic hydrolysis yield | 84.98 | | | | | |
| p= | | | | | | |
| Legend | | p < .01 | .01 <= p < .05 | .05 <= p < .10 | p >= .10 | |