

Supporting Information

Levulinic Acid Production from Furfural: Process Development and Techno-Economics

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Table S1: List of different reaction conditions used in this study for the conversion of FF/xylose to LA

Parameter tested (range)	Reagent, Reagent Concentration (g)	Solvent (g)		Temperature (°C)	Time (min)	Catalyst, weight (g)	Heating mode
		Water	Formalin				
Catalyst:FF loading (0.25:1 to 2:1 w/w)	FF, 0.25	2	0.5	170	60	1:1 Amberlyst-15:HZSM-5, -	MW
Reaction temperature (130 – 180 °C)	FF, 0.25	2	0.5	-	5 & 60	1:1 Amberlyst-15:HZSM-5, 0.5	MW
Reaction time (5 – 60 min)	FF, 0.25	2	0.5	170	-	1:1 Amberlyst-15:HZSM-5, 0.5	MW
Catalyst combination (0:1 to 1:0 w/w)	FF, 0.25	2	0.5	160	5	-, 0.5	MW
Formalin to FF ratio (1:1 to 5:1 w/w)	FF, 0.25	-	-	160	5	1:0 Amberlyst-15:HZSM-5, 0.5	MW
FF concentration (0 to 1.25 g)	FF, -	-	-	160	5	1:0 Amberlyst-15:HZSM-5, 0.5	MW
Conventional heating	FF, 0.25	2	0.5	160	5 & 60	-, 0.5	CH
Xylose to LA	Xylose, 0.25	2	0.5	160	5	1:0	MW

						Amberlyst-15:HZSM-5, 0.5
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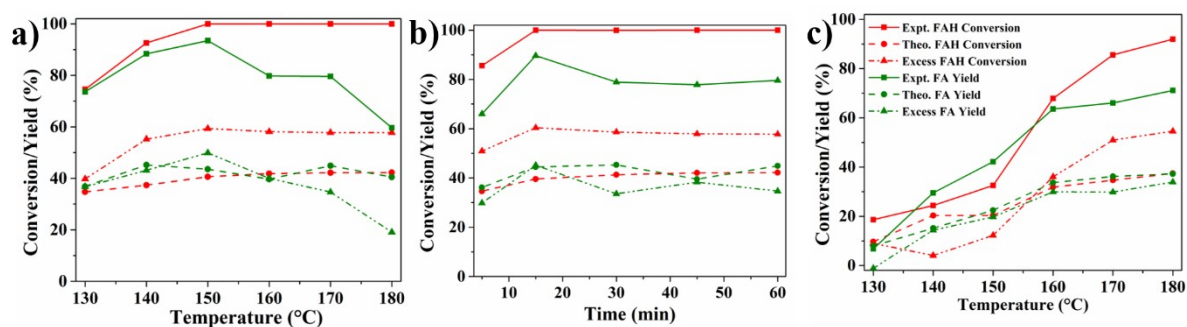


Figure S1: Theoretical and experimental FAH conversion and FA yields at varying a) temperature at 1h; b) time at 170°C and; c) temperature at 5 min (Reaction conditions: 2.5g solvent (formalin+water), 10 wt.% C_{FF} , formalin:catalyst:furfural=2:2:1 w/w/w, 2g water, catalyst= 1:1 Amberlyst-15:HZSM-5)

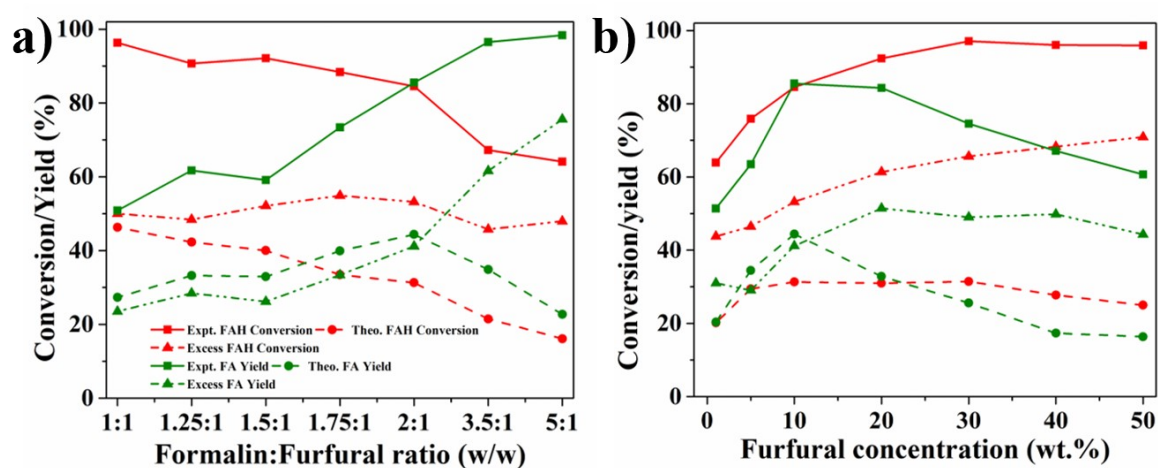


Figure S2: Theoretical and experimental FAH conversion and FA yields at varying a) formalin to furfural ratio; b) furfural concentration (C_{FF}) (Reaction conditions: 2.5g solvent(formalin+water), 5 min reaction time, 160°C reaction temperature, catalyst= 1:0 Amberlyst-15:HZSM-5)

Choice of organic solvent: It is essential to choose a suitable organic solvent to suppress the side product formation, especially from the furanic compounds such as HMF. Organic solvents such as dimethyl sulfoxide (DMSO), methyl isobutyl ketone (MIBK), and tetrahydrofuran (THF) were extensively used in the literature to suppress the by-product (humins) formation in converting C6 carbohydrates to platform chemicals.¹⁻³ Here, we utilized DMSO and MIBK as organic solvents in different proportions with water and studied their effect on the levulinic

acid production (*Figure S3*). The pure DMSO as organic solvent showed very low furfural conversion. The FF and FAH conversion was 4.0 and 2.0%, respectively, with HMF and LA yields as 3.6 and 1.1%. The yields of HMF, LA, and FF conversion were improved with water addition to DMSO in a 1:1 ratio. The side product formation was almost nullified and showed nearly 100% carbon balance. However, the conversions and yields were lower with DMSO than with the pure water as a solvent. This indicates that the DMSO suppresses the electrophilic substitution reaction along with the polymerization reactions of furanic compounds. The FF conversion was 67.8% and 52.8% with pure MIBK and MIBK+water, respectively. The LA yield in pure MIBK and MIBK+water mixture was 11.1 and 17.7%, with HMF yield of 0.2 and 5.2%, respectively. Where, the partition coefficient of FF, HMF, LA, and FA were 0.93, 0.48, 0.23, and 0.14 (mol/mL)/(mol/mL) respectively for the MIBK/water solvent system. Interestingly, complete conversion of FAH was observed in both cases with MIBK, which could be attributed to the formation of ketone-formaldehyde resins.⁴ Thus, MIBK cannot be the right organic solvent for this reaction system as it forms ketone-formaldehyde resins. This study reveals that the organic solvents were not effective in suppressing the polymerization reaction but interfere with the hydroxymethylation reaction to form HMF/LA.

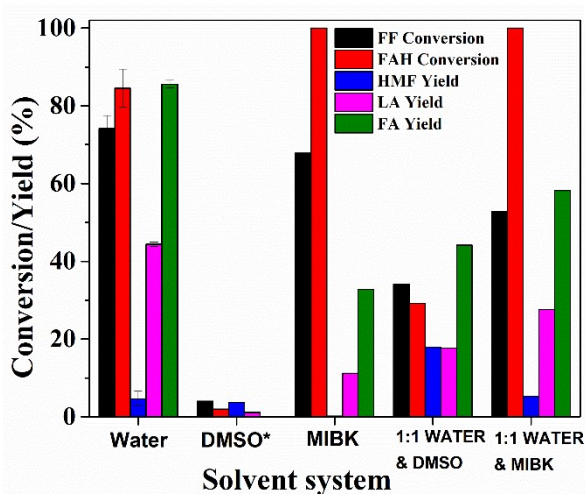


Figure S3: Role of solvents on the furfural conversion and levulinic acid (LA) production (reaction conditions: 2.5g solvent (formalin+water/DMSO/MIBK), 10 wt.% C_{FF} , catalyst:formalin:furfural= 2:2:1 w/w/w, 5 min reaction time, 160°C reaction temperature, catalyst= 1:0 Amberlyst-15:HZSM-5), * indicates catalyst:formalin:furfural= 2:1:1 w/w/w

Table S2: P-values from one-way ANOVA with conversion and yields as factor for variation of time at 170°C

Time (min)	P values
5	5.3631E-06
15	4.9981E-05
30	1.3506E-05
45	5.5592E-06
60	1.6913E-05

Table S3: P-values from one-way ANOVA with conversion and yields as factor for variation of catalyst loading

Catalyst loading (g)	P values
0.5	1.69134E-05
0.325	1.02669E-08
0.25	0.000442517
0.125	9.02109E-05
0.0625	0.017839437

Table S4: Energy consumption at various reaction temperatures

Catalyst	Reaction temperature (°C)	Energy consumption^a (KJ)
Amberlyst-15	150	34
	160	33
	170	55
HZSM-5	150	103
	160	106
	170	116

^a energy consumed was calculated for 5 min reaction time

Table S5: Comparison with various literature reported catalysts for the direct conversion to levulinic acid/ester

S.No	Reactant	Solvent	Catalyst	Heating source	Reaction conditions (Temp; Time; reactant; solvent)	Conversion (%)	Product selectivity (%)		Ref
							LA	MLE	
1	Furfural	Formalin (0.5g) /water (2.0g)	Amberlyst-15	MW	160°C; 5 min; 0.25g; 2.5g	74.1	59.9	-	This work

2	Furfural	Formalin (0.5g) /water (2.0g)	HZSM-5 (SAR-80)	MW	160°C; 5 min; 0.25g; 2.5g	71.2	19.7	-	This work
3	Xylose	Formalin (0.5g) /water (2.0g)	Amberlyst- 15	MW	160°C; 10 min; 0.25g; 2.5g	85.1	21.5	-	This work
4	Furfural	Formalin (0.5g) /water (2.0g)	1:1 Amberlyst- 15: HZSM-5	CH	160°C; 60 min; 0.25g; 2.5g	80.7	40.3	-	This work
5	Furfural	Formalin (0.5g) /water (2.0g)	HZSM-5 (SAR-60)	MW	170°C; 1h; 0.03g; 5g	90	46	-	5
6	Furfural	DMM /methanol	Amberlyst 70	CH	160°C; 2h; 1.8g; 40g	-	3.5 ^a	43.3 ^b	6
7	Furfural	DMM /methanol	D008 (acid resin)	CH	190°C; 1h; 4.14g; 60g	71.6	5.3	49.3	7
8	Furfural	DMM (20g)	Amberlyst- 15	CH	160°C; 2h; 2g; 20g	69.7	46.3	-	8
9	Furfural	DMM (10g) /water (10g)	Amberlyst- 15	CH	160°C; 2h; 2g; 20g	96.7	52.3	-	8
10	Furan	DMM (40g) /methanol (16g)	D008 (acid resin)	CH	170°C; 1.5h; 4g, 64g	97.2	69.8	-	9
11	Xylose	1,3,5-trioxane (0.42g)/ methanol (6g)	H-β zeolite	CH	160°C; 18h; 0.06g; 6.42g	-	-	47.4 ^b	10
12	Xylose	DMM /methanol	Amberlyst 70	CH	150°C; 6h; 1.8g; 40g	-	10.3 ^a	41.6 ^b	6
13	Xylose	DMM (20g)	Amberlyst- 15	CH	160°C; 2h; 2g; 20g	62.7	41.7	-	8
14	Furfural	DMM (10g) /water (10g)	Amberlyst- 15	CH	160°C; 2h; 2g; 20g	95.8	47.4	-	8
15	Bamboo	DMM (25g) /methanol (25g)	C ₇ H ₇ - SO ₃ H	CH	200°C; 2h; 5g; 50g	81.3	-	50.6	11
16	Bamboo	DMM (25g) /methanol (25g)	ZSM-5	CH	200°C; 2h; 5g; 50g	37.4	-	27.7	11

17	Bagasse	DMM (10g) /ethanol (10g)	H- β zeolite	CH	200°C; 4h; 2g; 20g	54.2	-	24.4 ^c	12
18	Rice straw	DMM (15g) /methanol (25g)	Amberlyst- 15	CH	200°; 1h; 4g; 40g	83.5	-	38.9	13
MW- Microwave heating; CH- convectional heating; ^a yield of LA; ^b yield of MLE; ^c ELE: Ethyl levulinate									

Table S6: moles of formaldehyde consumed and formic acid formed at varying formalin to furfural ratio

Formalin: furfural ratio	Initial moles of FAH	Initial moles of FF	Moles of FAH consumed	Moles of FA formed
1:1	0.00315	0.00269	0.00303	0.00140
1.25:1	0.00398		0.00361	0.00164
1.5:1	0.00465		0.00429	0.00156
1.75:1	0.00542		0.00479	0.00201
2:1	0.00621		0.00555	0.00218
3.5:1	0.01076		0.00723	0.00272
5:1	0.01593		0.00993	0.00313

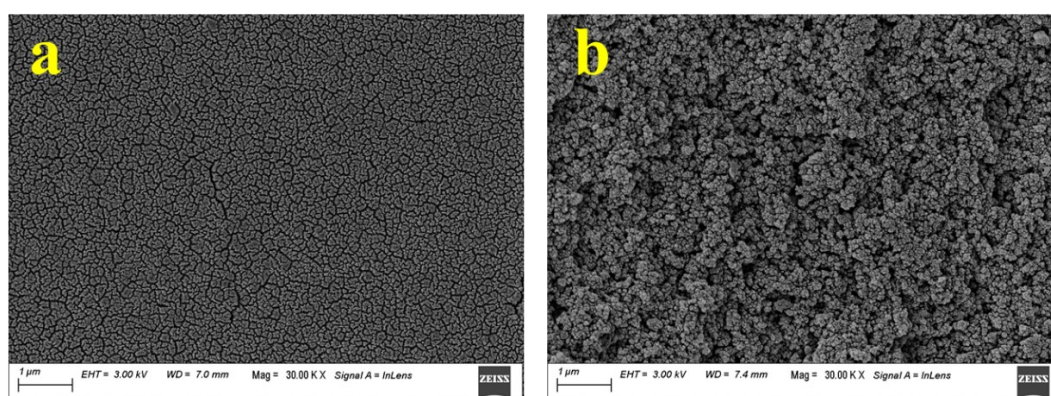


Figure S4: FESEM images of a) fresh and b) spent catalyst (1:0 Amberlyst-15: HZSM-5)

Table S7: List of assumptions and economic considerations

Furfural price (\$/kg) ^a	1
Formalin price (\$/kg) ^a	0.69
Xylose price (\$/kg) ^a	0.23
Cellulose price (\$/kg) ^a	0.69
Biomass price (\$/kg) ^a	0.07

Amberlyst-15 catalyst price (\$/kg) ^a	21
HMF price (\$/kg) ^a	5
FA price (\$/kg) ^a	0.652
Cooling water (\$/kJ) ¹⁴	2.12e-7
Electricity price(\$/kWh)	0.08
Plant life (year) ¹⁵	30
Working days per year ¹⁶	334
Tax rate (%)	18
Discount rate (%) ¹⁵	10
Interest rate (%)	10
Financing	100% equity
Working capital	5% FCC
Plant depreciation	200% declining balance
Plant recovery period	7 years

^acost of chemicals are taken from the world largest bulk supplier (Alibaba.com)

Table S8: Experimental and normalized weights of reactants and products

	Initial weights fed to reactor (kg)	Final converted/formed weights after reaction (kg)	Normalized weights (kg)
Furfural	100.0	74.1	1000
Formaldehyde	74.0	62.5	843
Water	800.0	800.0	10796
Levulinic acid		53.6	723
Formic acid		41.0	553
HMF		6.1	82
Humins		21.6	291

Table S9: Experimental weights of reactants and products feed/obtained at each reactor in the multi-step conversion of biomass to LA

	Biomass	FAH	Xylose	Hydrochar	Cellulose	LA	FF	HMF	FA (kg)	Ref
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	(kg)	(kg)	(kg)	(kg)	(kg)	(kg)	(kg)	(kg)		
PTU (Biomass to Xylose and HC)	100		19.6	61.6	31					17
BR (HC to LA)						12.3		1	5.8	18
ER (Xylose to FF)							9.7			19
MWR (FF to LA)		8.2				7.0		0.8	5.3	this work
Total In/out	100	8.2				19.2		1.8	11.2	

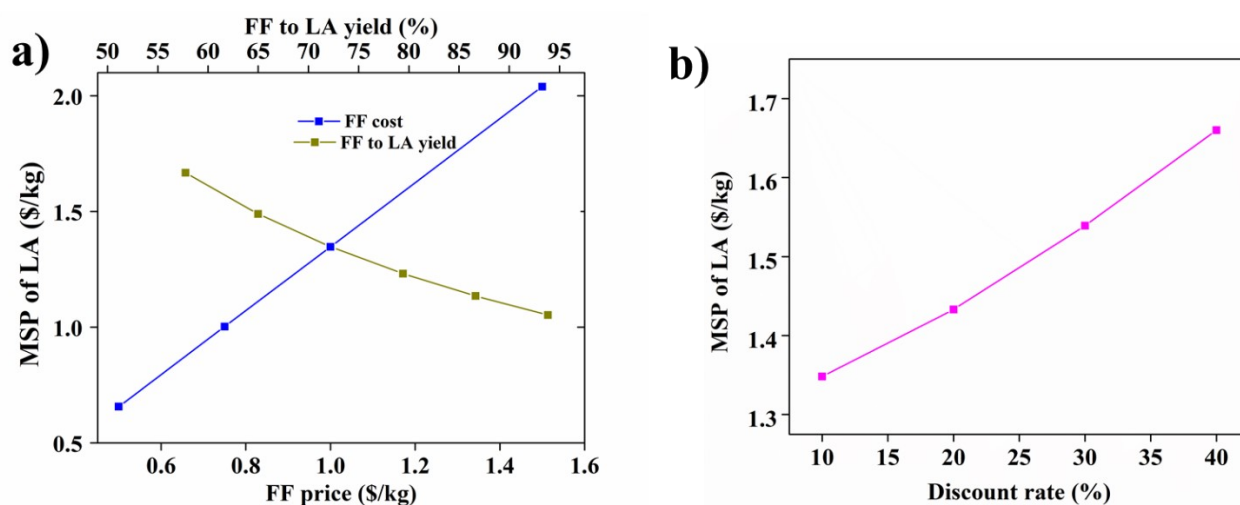


Figure S5: a) Effect of furfural (FF) price and plant efficiency, b) discount rate on the MSP of LA (produced from furfural)

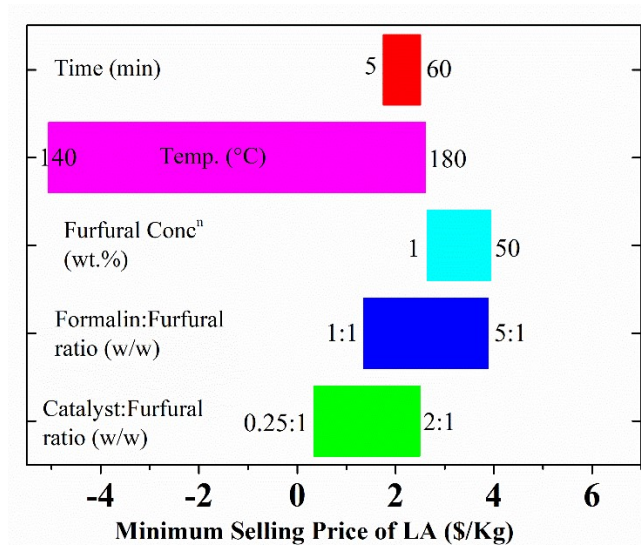


Figure S6: Sensitivity analysis of MSP of LA (produced from FF) with varying reaction parameters

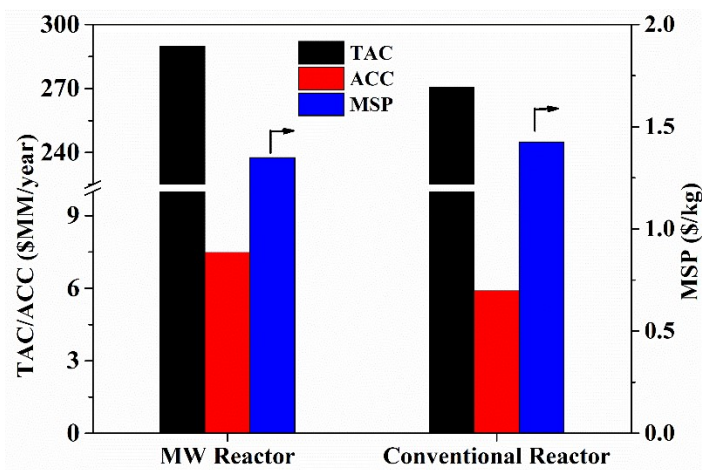


Figure S7: Comparison of MSP's obtained from Microwave (MW) and Conventional reactors (Reaction conditions: 2.5g solvent (formalin+water), 10 wt.% C_{FF} , catalyst:formalin:furfural=2:2:1 w/w/w, 2g water, 160°C reaction temperature, 5 min reaction time for microwave reactor and 60 min for convectional reactor)

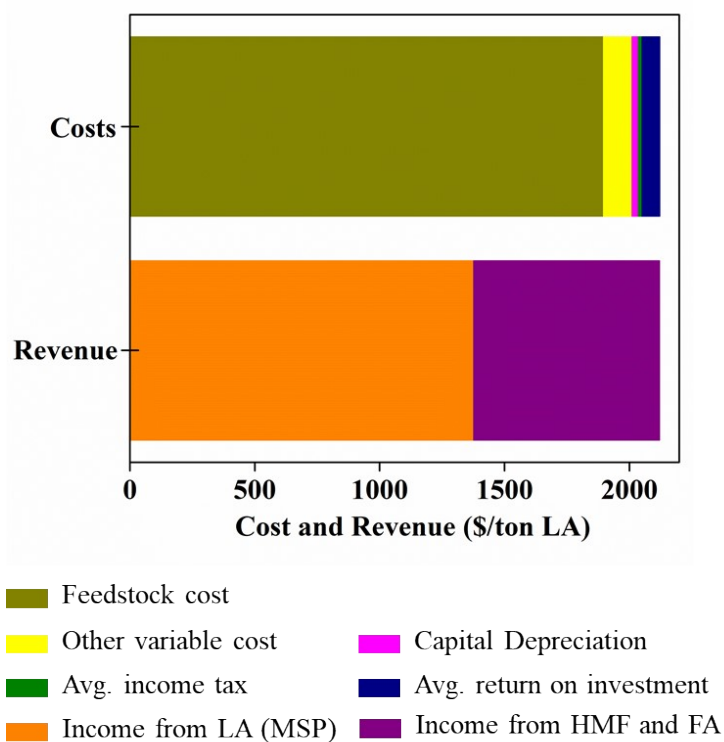


Figure S8: Cost and revenue accounted for minimum selling price (MSP) of LA produced from cellulose

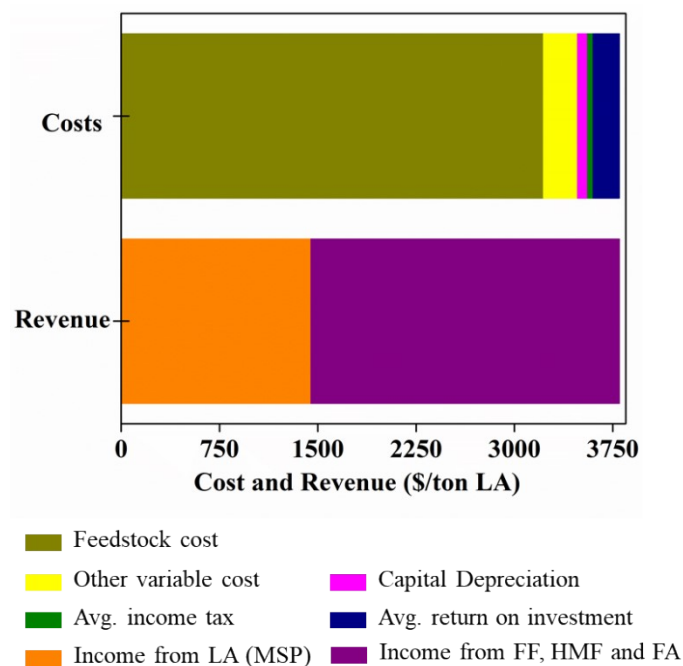


Figure S9: Cost and revenue responsible for the MSP of LA (produced from xylose)

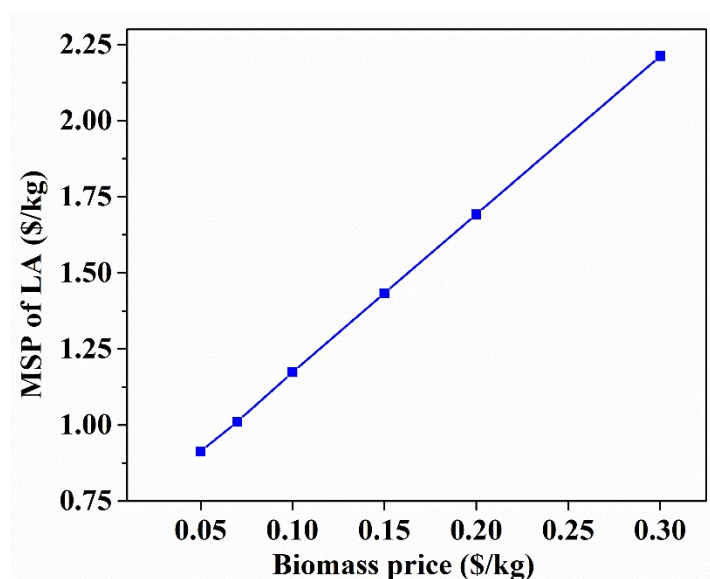


Figure S10: Effect of biomass price on the MSP of LA

Table S10: Detailed Purchased/installed costs of equipment's, Total direct cost, Total indirect cost, fixed capital cost and total capital cost for production of LA from furfural in a microwave reactor

Process section		Purchased cost (\$MM)	Installed cost (\$MM)
LA production	MWR/BR	7.86	11.32
	PTU	0.10	0.14
	HE	0.20	0.29

	Sub-Total	8.16	11.75
	SLS	0.36	0.518
LA separation	EC & DC ²⁰	9.4	13.54
	Sub-Total	9.76	14.05
Waste water treatment ²¹		4.37	6.29
Onsite storage		0.6	1.02
Utilities ²¹		1.9	3.23
ISBL Total ^a		17.92	25.80
Total		24.79	36.35
Warehouse	4% ISBL		1.03
Site development	9% ISBL		2.32
Additional piping	4.5% ISBL		1.16
Total Direct Cost			40.86
Prorateable expenses	10% TDC		4.09
Field expenses	10% TDC		4.09
Home office & construction fee	20% TDC		8.17
Project contingency	10% TDC		4.09
Other costs	10% TDC		4.09
Total indirect cost			24.52
Fixed capital cost			65.38
Land			1.80
Working capital	5% FCC		3.27
Total capital cost			70.45
Lang factor (FCC/purchased capital cost ^b)		3.20	

^aISBL includes LA production and separation costs only

^bwaste water treatment plant cost was not included in calculation of lang factor

Table S11: Discounted cash flow rate of return worksheet

Year	-2	-1	0	1	2	3	4	5	6
Fixed capital investment (\$)	52,30,400	3,92,28,000	2,09,21,600						
land (\$)	18,48,000								
working capital (\$)			32,69,000						
fuel sales (\$/yr)				12,21,02,277	16,28,03,036	16,28,03,036	16,28,03,036	16,28,03,036	16,28,03,036
by product credit (\$/yr)				9,66,41,931	12,88,55,908	12,88,55,908	12,88,55,908	12,88,55,908	12,88,55,908
total annual sales (\$/yr)				21,87,44,208	29,16,58,944	29,16,58,944	29,16,58,944	29,16,58,944	29,16,58,944
annual manufacturing cost									
feedstock (\$/yr)				20,62,52,124	27,50,02,832	27,50,02,832	27,50,02,832	27,50,02,832	27,50,02,832
baghouse bags (\$/yr)				5,19,687					5,19,687
other variable costs (\$/yr)				56,34,750	75,13,000	75,13,000	75,13,000	75,13,000	75,13,000
total product cost (\$/yr)				21,24,06,561	28,25,15,832	28,25,15,832	28,25,15,832	28,25,15,832	28,30,35,519
annual depreciation									
depreciation charges (\$/yr)				93,42,802	1,60,11,562	1,14,34,962	81,65,962	58,38,434	58,31,896
remaining value				5,60,37,198	4,00,25,636	2,85,90,674	2,04,24,712	1,45,86,278	87,54,382
Net revenue (\$/yr)				-30,05,155	-68,68,450	-22,91,850	9,77,150	33,04,678	27,91,529
losses forwarded (\$/yr)					-30,05,155	-98,73,605	-1,21,65,455	-1,11,88,305	-78,83,628
taxable income (\$/yr)				-30,05,155	-98,73,605	-1,21,65,455	-1,11,88,305	-78,83,628	-50,92,099
income tax (\$/yr)				-	-	-	-	-	-
annual cash income (\$/yr)				63,37,647	91,43,112	91,43,112	91,43,112	91,43,112	86,23,425
discount factor	1.21	1.10	1.00	0.91	0.83	0.75	0.68	0.62	0.56
annual present value (\$/yr)				57,61,497	75,56,291	68,69,355	62,44,868	56,77,153	48,67,699
total capital investment + interest	8564864	43150800	24190600						
Net Present worth			-1388						

Year	7	8	9	10	11	12	13	14	15
Fixed capital investment (\$)									
land (\$)									
working capital (\$)									
fuel sales (\$/yr)	16,28,03,036	16,28,03,036	16,28,03,036	16,28,03,036	16,28,03,036	16,28,03,036	16,28,03,036	16,28,03,036	16,28,03,036
by product credit (\$/yr)	12,88,55,908	12,88,55,908	12,88,55,908	12,88,55,908	12,88,55,908	12,88,55,908	12,88,55,908	12,88,55,908	12,88,55,908
total annual sales (\$/yr)	29,16,58,944	29,16,58,944	29,16,58,944	29,16,58,944	29,16,58,944	29,16,58,944	29,16,58,944	29,16,58,944	29,16,58,944
annual manufacturing cost									
feedstock (\$/yr)	27,50,02,831	27,50,02,831	27,50,02,831	27,50,02,831	27,50,02,831	27,50,02,831	27,50,02,831	27,50,02,831	27,50,02,831
baghouse bags (\$/yr)					5,19,687				
other variable costs (\$/yr)	75,13,000	75,13,000	75,13,000	75,13,000	75,13,000	75,13,000	75,13,000	75,13,000	75,13,000
total product cost (\$/yr)	28,25,15,831	28,25,15,831	28,25,15,831	28,25,15,831	28,30,35,518	28,25,15,831	28,25,15,831	28,25,15,831	28,25,15,831
annual depreciation charges (\$/yr)	58,38,434	29,15,948							
remaining value	29,15,948	-0							
Net revenue (\$/yr)	33,04,678	62,27,164	91,43,112	91,43,112	86,23,425	91,43,112	91,43,112	91,43,112	91,43,112
losses forwarded (\$/yr)	-50,92,099	-17,87,421	-	-	-	-	-	-	-
taxable income (\$/yr)	-17,87,421	44,39,743	91,43,112	91,43,112	86,23,425	91,43,112	91,43,112	91,43,112	91,43,112
income tax (\$/yr)	-	7,99,154	16,45,760	16,45,760	15,52,216	16,45,760	16,45,760	16,45,760	16,45,760
annual cash income (\$/yr)	91,43,112	83,43,958	74,97,352	74,97,352	70,71,208	74,97,352	74,97,352	74,97,352	74,97,352
discount factor	0.51	0.47	0.42	0.39	0.35	0.32	0.29	0.26	0.24
annual present value (\$/yr)	46,91,862	38,92,518	31,79,609	28,90,554	24,78,415	23,88,887	21,71,716	19,74,287	17,94,806
total capital investment + interest									
Net Present worth									
Year	16	17	18	19	20	21	22	23	24
Fixed capital									

investment (\$)									
land (\$)									
working capital (\$)									
fuel sales (\$/yr)	16,28,03,036	16,28,03,036	16,28,03,036	16,28,03,036	16,28,03,036	16,28,03,036	16,28,03,036	16,28,03,036	16,28,03,036
by product credit (\$/yr)	12,88,55,908	12,88,55,908	12,88,55,908	12,88,55,908	12,88,55,908	12,88,55,908	12,88,55,908	12,88,55,908	12,88,55,908
total annual sales (\$/yr)	29,16,58,944	29,16,58,944	29,16,58,944	29,16,58,944	29,16,58,944	29,16,58,944	29,16,58,944	29,16,58,944	29,16,58,944
annual manufacturing cost feedstock (\$/yr)	27,50,02,831	27,50,02,831	27,50,02,831	27,50,02,831	27,50,02,831	27,50,02,831	27,50,02,831	27,50,02,831	27,50,02,831
baghouse bags (\$/yr)	5,19,687					5,19,687			
other variable costs (\$/yr)	75,13,000	75,13,000	75,13,000	75,13,000	75,13,000	75,13,000	75,13,000	75,13,000	75,13,000
total product cost (\$/yr)	28,30,35,518	28,25,15,831	28,25,15,831	28,25,15,831	28,25,15,831	28,30,35,518	28,25,15,831	28,25,15,831	28,25,15,831
annual depreciation depreciation charges (\$/yr)									
remaining value									
Net revenue (\$/yr)	86,23,425	91,43,112	91,43,112	91,43,112	91,43,112	86,23,425	91,43,112	91,43,112	91,43,112
losses forwarded (\$/yr)	-	-	-	-	-	-	-	-	-
taxable income (\$/yr)	86,23,425	91,43,112	91,43,112	91,43,112	91,43,112	86,23,425	91,43,112	91,43,112	91,43,112
income tax (\$/yr)	15,52,216	16,45,760	16,45,760	16,45,760	16,45,760	15,52,216	16,45,760	16,45,760	16,45,760
annual cash income (\$/yr)	70,71,208	74,97,352	74,97,352	74,97,352	74,97,352	70,71,208	74,97,352	74,97,352	74,97,352
discount factor	0.22	0.20	0.18	0.16	0.15	0.14	0.12	0.11	0.10
annual present value (\$/yr)	15,38,901	14,83,311	13,48,465	12,25,877	11,14,434	9,55,536	9,21,019	8,37,290	7,61,173
total capital investment + interest									
Net Present worth									

Year	25	26	27	28	29	30
Fixed capital investment (\$)						
land (\$)						
working capital (\$)						
fuel sales (\$/yr)	16,28,03,036	16,28,03,036	16,28,03,036	16,28,03,036	16,28,03,036	16,28,03,036
by product credit (\$/yr)	12,88,55,908	12,88,55,908	12,88,55,908	12,88,55,908	12,88,55,908	12,88,55,908
total annual sales (\$/yr)	29,16,58,944	29,16,58,944	29,16,58,944	29,16,58,944	29,16,58,944	29,16,58,944
annual manufacturing cost						
feedstock (\$/yr)	27,50,02,831	27,50,02,831	27,50,02,831	27,50,02,831	27,50,02,831	27,50,02,831
baghouse bags (\$/yr)		5,19,687				
other variable costs (\$/yr)	75,13,000	75,13,000	75,13,000	75,13,000	75,13,000	75,13,000
total product cost (\$/yr)	28,25,15,831	28,30,35,518	28,25,15,831	28,25,15,831	28,25,15,831	28,25,15,831
annual depreciation						
depreciation charges (\$/yr)						
remaining value						
Net revenue (\$/yr)	91,43,112	86,23,425	91,43,112	91,43,112	91,43,112	91,43,112
losses forwarded (\$/yr)	-	-	-	-	-	-
taxable income (\$/yr)	91,43,112	86,23,425	91,43,112	91,43,112	91,43,112	91,43,112
income tax (\$/yr)	16,45,760	15,52,216	16,45,760	16,45,760	16,45,760	16,45,760
annual cash income (\$/yr)	74,97,352	70,71,208	74,97,352	74,97,352	74,97,352	74,97,352
discount factor	0.09	0.08	0.08	0.07	0.06	0.06
annual present value (\$/yr)	6,91,976	5,93,313	5,71,881	5,19,891	4,72,629	4,29,662
total capital investment + interest						
Net Present worth						

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