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APPENDICES

Appendix A Calculation Feed Flowrate for Steaming and Activity Testing

The temperature and vapor pressure of steaming conditions were varied from the selected nitrogen gas flow rate.

Table A1 Calculation of water flowrate for steaming treatment

Condition	Temperature (°C)	Vapor pressure (kPa)	N_2 ($mol \cdot h^{-1}$)	H_2O ($mol \cdot h^{-1}$)	WHSV (h^{-1})
ZP5	400	5.00	0.1087	0.0056	0.1016
ZP20	400	20.00	0.1087	0.0267	0.4813
ZP40	400	40.00	0.1087	0.0709	1.2766
ZT350	350	26.75	0.1174	0.0421	0.7583
ZT500	500	26.75	0.0946	0.0340	0.6112
ZT650	650	26.75	0.0792	0.0284	0.5118

Fixed: N_2 flowrate = 100 mL·min = 6000 mL·h

At 400 °C, vapor pressure 5 kPa, and operating pressure 1 atm

Density of N_2 = $0.0005072\ g \cdot mL^{-1}$

$$\text{Mole of } N_2 = \frac{(6000\ mL \cdot h^{-1}) \times (0.0005072\ g \cdot mL^{-1})}{(28\ g \cdot mol^{-1})}$$

$$= 0.1087\ mol \cdot h^{-1}$$

Total mole = H_2O mole + N_2 mole

Mole fraction = Pressure fraction

$$\frac{H_2O}{0.1087 + H_2O} = \frac{5\ kPa}{101.3\ kPa}$$

H_2O = $0.0056\ mol \cdot h^{-1}$

$$(0.0056\ mol \cdot h^{-1}) \times (18\ g \cdot mol^{-1})$$

$$= 0.1016\ g \cdot h^{-1}$$

Calculation of n-pentane feed flow rate at WHSV = 5 h^{-1}

Amount of HZSM-5 catalyst = 0.20 g

$$\text{WHSV} = \frac{\text{Flow rate } (g \cdot h^{-1})}{\text{Weight of catalyst}}$$

$$5\ h^{-1} = \frac{\text{Flow rate } (g \cdot h^{-1})}{0.20\ g}$$

$$\text{Flow rate} = 1.0\ g \cdot h^{-1}$$

According to n-pentane density is equal to 0.626 g·mL at 20 °C, 1 atm

$$\text{Flow rate} = \frac{1.0\ g \cdot h^{-1}}{0.626\ g \cdot mL}$$

$$= 1.597\ mL \cdot h^{-1}$$

Calculation of light naphtha (75 $\%C_5$ and 25 $\%C_6$) feed flow rate at WHSV = 2 h^{-1}

Amount of HZSM-5 catalyst = 0.20 g

$$\text{WHSV} = \frac{\text{Flow rate } (g \cdot h^{-1})}{\text{Weight of catalyst}}$$

$$2\ h^{-1} = \frac{\text{Flow rate } (g \cdot h^{-1})}{0.20\ g}$$

$$\text{Flow rate} = 0.4\ g \cdot h^{-1}$$

According to light naphtha density is equal to 0.634 g·mL at 20 °C, 1 atm

$$\text{Flow rate} = \frac{0.4\ g \cdot h^{-1}}{0.634\ g \cdot mL}$$

$$= 0.631\ mL \cdot h^{-1}$$

Appendix B Mass Balance Calculation of CLD/Ga/Ac/ZP5 Catalyst

Condition:	Pressure	=	1	atm
	Temperature	=	500	°C
	Catalyst amount	=	0.2	g
	WHSV	=	5	h^{-1}
		=	Feed flowrate / amount of catalyst	
	Feed flowrate	=	$5\ h^{-1} \times 0.2\ g$	
		=	$0.1\ g\cdot h^{-1}$	

At 20 °C and 1 atm, the density of *n*-pentane is $0.626\ g\cdot mL^{-1}$

n-Pentane feed rate	=	$(0.1\ g\cdot h^{-1}) / (0.626\ g\cdot mL^{-1})$	
	=	$1.597\ mL\cdot h^{-1}$	
Nitrogen carrier gas	=	25.4	$mL\cdot min^{-1}$
	=	$25.4\ mL\cdot min^{-1} \times (0.001165\ g\cdot mL^{-1})$	
	=	$0.0296\ g\cdot min^{-1}$	

Density properties:	*n*-Pentane	=	0.626	$g\cdot mL^{-1}$
	Hydrogen	=	0.000083	$g\cdot mL^{-1}$
	Nitrogen	=	0.001165	$g\cdot mL^{-1}$

Table B1 Volume of product from wet test equipment

TOS (min)	N_2+H_2+HC (mL)	N_2 (mL)	Duration (min)	Area H_2	H_2 (mL)	HC (mL)
80	1924	1524	60	30722	127	273.3
140	1972	1524	60	37093	157	291.2
200	1942	1524	60	37734	157	260.9
260	1783	1524	60	34064	130	128.8

Note: The area of pulsing H_2 1 mL is equal to 466456

Example calculate at TOS = 80 min

N_2 volume = (25.4 mL·min^{-1}) × 60 min = 1524 mL

H_2 volume = (30722/466456) × 1924 mL= 127 mL

HC volume = 1924 – 1524 – 127 = 273.3 mL

Table B2 Product selectivity and conversion of *n*-pentane over CLD/Ga/Ac/ZP5 catalyst (Reaction condition: 500 °C, 1 atm, and WHSV = 5 h-1)

TOS (min)	80	140	200	260
Conversion	88.30	83.75	81.40	80.97
Aromatic selectivity	93.68	90.00	91.30	90.20
Light hydrocarbon selectivity	6.32	10.00	8.70	9.80
Benzene selectivity	6.51	5.63	15.76	10.49
Toluene selectivity	52.98	44.06	45.11	45.18
Ethylbenzene selectivity	3.36	4.15	2.46	3.09
Xylene selectivity	30.83	36.16	27.96	31.44
Xylene yield	27.23	30.28	22.76	25.46
p-Xylene	19.57	21.28	16.46	18.55
m-Xylene	7.09	8.56	5.96	6.53
o-Xylene	0.57	0.44	0.34	0.37
p-Xylene selectivity in xylenes	71.88	70.27	72.32	72.87
Overall mass balance error (%)	2.20	1.72	2.11	2.14
Carbon balance error (%)	13.21	10.66	0.55	0.55
Hydrogen balance error (%)	29.38	24.69	0.00	0.00

Overall Balance at TOS = 80 min

Component	Input(g)	Component	Output(g)
n-Pentane	1.000	Gas Product	1.051
Nitrogen	1.785	Nitrogen	1.785
Hydrogen	0.005	Hydrogen	0.011
Total	2.785	Total	2.847

Compound	Area	%Area=%Wt	MW	Mole	%Mole = %Vol	Vol (mL)	Mass (g)	Wt. C (g)	Wt. H (g)
Ethane	1	0.0000	30	0.0000	0.00	0.00	0.00	0.0000	0.0000
Ethylene	841	0.0044	28	0.0002	1.35	3.70	0.00	0.0040	0.0007
Propane	2,338	0.0121	44	0.0003	2.40	6.55	0.01	0.0105	0.0023
Propylene	2,618	0.0136	42	0.0003	2.81	7.68	0.01	0.0123	0.0021
Butane	2,710	0.0141	58	0.0002	2.11	5.76	0.01	0.0123	0.0026
Acetylene	323	0.0017	26	0.0001	0.56	1.53	0.00	0.0016	0.0001
1-Butene	779	0.0040	56	0.0001	0.63	1.71	0.00	0.0037	0.0006
i-Butene	816	0.0042	56	0.0001	0.66	1.79	0.00	0.0038	0.0006
n-Pentane	22,560	0.1172	72	0.0016	14.13	38.61	0.12	0.1034	0.0207
Benzene	11,074	0.0575	78	0.0007	6.40	17.49	0.06	0.0562	0.0047
Toluene	90,177	0.4686	92	0.0051	44.20	120.78	0.50	0.4529	0.0431
Ethylbenzene	5,726	0.0298	106	0.0003	2.44	6.66	0.03	0.0285	0.0030
p-, *m*-Xylene	51,389	0.2670	106	0.0025	21.86	59.74	0.28	0.2560	0.0267
o-Xylene	1,096	0.0057	106	0.0001	0.47	1.27	0.01	0.0055	0.0006
Total	192,448	1.0000		0.0115	100.00	273.28	1.06	0.9509	0.1077

Overall Balance at TOS = 140 min

Component	Input(g)	Component	Output(g)
n-Pentane	1.000	Gas Product	1.055
Nitrogen	1.775	Nitrogen	1.775
Hydrogen	0.000	Hydrogen	0.013
Total	2.775	Total	2.844

Compound	Area	%Area=%Wt	MW	Mole	%Mole = %Vol	Vol (mL)	Mass (g)	Wt. C (g)	Wt. H (g)
Methane	1,045.3	0.0049	16	0.00	2.44	7.12	0.01	0.0038	0.0013
Ethane	2,958.2	0.0139	30	0.00	3.69	10.75	0.01	0.0115	0.0029
Ethylene	4,307.6	0.0203	28	0.00	5.76	16.76	0.02	0.0180	0.0030
Propane	4,372.7	0.0206	44	0.00	3.72	10.83	0.02	0.0174	0.0039
Propylene	1,347.0	0.0063	42	0.00	1.20	3.49	0.01	0.0056	0.0009
Butane	1,831.8	0.0086	58	0.00	1.18	3.44	0.01	0.0074	0.0015
Acetylene	846.3	0.0040	26	0.00	1.22	3.55	0.00	0.0038	0.0003
1-Butene	483.8	0.0023	56	0.00	0.32	0.94	0.00	0.0020	0.0003
i-Butene	620.8	0.0029	56	0.00	0.41	1.21	0.00	0.0026	0.0004
n-Pentane	34,560.2	0.1625	72	0.00	17.96	52.31	0.17	0.1401	0.0280
Benzene	10,037.2	0.0472	78	0.00	4.82	14.02	0.05	0.0451	0.0038
Toluene	78,476.2	0.3690	92	0.00	31.92	92.95	0.38	0.3486	0.0332
Ethylbenzene	7,392.5	0.0348	106	0.00	2.61	7.60	0.04	0.0326	0.0034
p-, *m*-Xylene	63,467.2	0.2984	106	0.00	22.41	65.25	0.31	0.2796	0.0291
o-Xylene	939.7	0.0044	106	0.00	0.33	0.97	0.00	0.0041	0.0004
Total	212,686.5	1.0000		0.01	100.00	291.18	1.03	0.9222	0.1125

Overall Balance at TOS = 200 min

Component	Input(g)	Component	Output(g)
n-Pentane	1.000	Gas Product	0.937
Nitrogen	1.775	Nitrogen	1.775
Hydrogen	0.000	Hydrogen	0.013
Total	2.775	Total	2.726

Compound	Area	%Area=%Wt	MW	Mole	%Mole=%Vol	Vol (mL)	Mass (g)	Wt. C (g)	Wt. H (g)
Methane	469.5	0.24	16	0.02	1.20	3.13	0.00	0.0017	0.0006
Ethane	1,030.3	0.53	30	0.02	1.40	3.66	0.00	0.0039	0.0010
Ethylene	2,835.2	1.45	28	0.05	4.14	10.80	0.01	0.0116	0.0019
Propane	2,660.4	1.36	44	0.03	2.47	6.45	0.01	0.0104	0.0023
Propylene	4,293.9	2.20	42	0.05	4.18	10.91	0.02	0.0175	0.0029
Butane	802.7	0.41	58	0.01	0.57	1.48	0.00	0.0032	0.0007
Acetylene	768.8	0.39	26	0.02	1.21	3.15	0.00	0.0034	0.0003
1-Butene	558.5	0.29	56	0.01	0.41	1.06	0.00	0.0023	0.0004
i-Butene	391.8	0.20	56	0.00	0.29	0.75	0.00	0.0016	0.0003
n-Pentane	36,249.1	18.60	72	0.26	20.59	53.71	0.17	0.1439	0.0288
Benzene	25,010.3	12.83	78	0.16	13.11	34.21	0.12	0.1100	0.0092
Toluene	71,576.5	36.72	92	0.40	31.81	83.01	0.34	0.3113	0.0296
Ethylbenzene	3,907.1	2.00	106	0.02	1.51	3.93	0.02	0.0169	0.0018
p-, *m*-Xylene	43,685.6	22.41	106	0.21	16.85	43.97	0.21	0.1884	0.0196
o-Xylene	667.9	0.34	106	0.00	0.26	0.67	0.00	0.0029	0.0003
Total	194,907.6	100.00		1.25	100.00	260.90	0.93	0.8288	0.0996

Overall Balance at TOS = 260 min

Component	Input(g)	Component	Output(g)
n-Pentane	1.0000	Gas Product	0.9298
Nitrogen	1.7755	Nitrogen	1.7755
Hydrogen	0	Hydrogen	0.0108
Total	2.7755	Total	2.7161

Compound	Area	%Area=%Wt.	MW	Mole	%Mole = %Vol	Vol (mL)	Mass (g)	Wt. C (g)	Wt. H (g)
Methane	411.4	0.21	16	0.01	1.05	1.35	0.00	0.0007	0.0002
Ethane	1,125.1	0.58	30	0.02	1.53	4.00	0.01	0.0043	0.0011
Ethylene	2,431.0	1.25	28	0.04	3.55	9.27	0.01	0.0099	0.0017
Propane	3,768.2	1.93	44	0.04	3.50	9.14	0.02	0.0147	0.0033
Propylene	3,902.2	2.00	42	0.05	3.80	9.92	0.02	0.0159	0.0027
Butane	987.2	0.51	58	0.01	0.70	1.82	0.00	0.0039	0.0008
Acetylene	1,283.7	0.66	26	0.03	2.02	5.27	0.01	0.0056	0.0005
1-Butene	720.7	0.37	56	0.01	0.53	1.37	0.00	0.0029	0.0005
i-Butene	871.0	0.45	56	0.01	0.64	1.66	0.00	0.0036	0.0006
n-Pentane	37,172.4	19.07	72	0.26	21.12	55.11	0.18	0.1476	0.0295
Benzene	16,584.7	8.51	78	0.11	8.70	22.70	0.08	0.0729	0.0061
Toluene	71,456.9	36.66	92	0.40	31.78	82.90	0.34	0.3109	0.0296
Ethylbenzene	4,888.1	2.51	106	0.02	1.89	4.92	0.02	0.0211	0.0022
p-, *m*-Xylene	49,003.9	25.14	106	0.24	18.91	49.34	0.23	0.2115	0.0220
o-Xylene	730.6	0.37	106	0.00	0.28	0.74	0.00	0.0032	0.0003
Total	195,337.0	100.22		1.25	100.00	259.51	0.93	0.8288	0.1010

Appendix C The Gallium Contents Before and After Activity Testing

At atmospheric pressure, the metallic gallium is melted at 29.76 °C and vaporized at 2200 °C. The gallium has a low vapor pressure at high temperatures. During reduction, Ga_2O_3 species was converted to Ga_2O and gallium (III) hydride compounds. It was speculated that gallium (III) hydride could transform to be the metallic gallium forms resulting in vaporization of gallium metal during reaction. Therefore, the measurement of Ga species in spent catalysts will allow for the determination of Ga loss after the reaction, if any. Table A3 shows that the gallium is unchanged at 0.70 wt% after reaction at 500 °C. This result was consistent with that of Tagliabue and co-worker in 2004 reporting that gallium content was no significant loss of trivalent element during the reaction and thermal treatments.

Table A3 The gallium content in fresh and spent CLD/Ga/Ac/ZP5 catalysts by XRF

Catalysts	SiO_2 (wt%)	Al_2O_3 (wt%)	Ga (wt%)
CLD/Ga/Ac/ZP5	92.56	6.74	0.70
Spent CLD/Ga/Ac/ZP5	93.16	6.14	0.70

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1. Thanatawee, P.; Jongpatiwut, S.; Rirksomboon, T.; and Kitiyanan, B. (2015, April 21) Influences of Catalyst Formulation on the Catalytic Activity of Modified HZSM-5 in the Aromatization of Light Paraffins. Proceeding of the 6th Research Symposium on Petrochemicals and Materials Technology and the 21st PPC Symposium on Petroleum, Petrochemicals, and Polymers 2015, Bangkok, Thailand.

Presentations:

1. Thanatawee, P.; Jongpatiwut, S.; Rirksomboon, T.; and Kitiyanan, B. (2015, June 21-24) Mild steaming on modified HZSM-5 for the aromatization of light paraffins. Paper presented at 5th International Colloids Conference 2015, Amsterdam, Netherlands.
2. Thanatawee, P.; Jongpatiwut, S.; Rirksomboon, T.; and Kitiyanan, B. (2015, April 21) Influences of Catalyst Formulation on the Catalytic Activity of Modified HZSM-5 in the Aromatization of Light Paraffins. Paper presented at The 6th Research Symposium on Petrochemicals and Materials Technology and the 21st PPC Symposium on Petroleum, Petrochemicals, and Polymers 2015, Bangkok, Thailand.