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APPENDICES

Appendix A Calculation of %weight of Pd metal loaded on TUD-1 via impregnation technique

Thisimpregnation techniqueuses 0.30 g of TUD-1 support, 3 ml of methanol and various amounts of Pdspecies derived from $Pd(NO_3)_2$solution.

The following equation is used to calculate the weight (in gram) of Pdused for preparing Pd-TUD-1 with different Pd concentration on 0.3g of TUD-1 support.

$$\%\text{ weight of Pd on 0.3-g TUD-1} = \frac{\text{weight (g) of Pd}}{\text{weight (g) of Pd} + 0.3\text{ g of TUD-1 support}} \times 100\%$$

$$= A$$

Since the $Pd(NO_3)_2$ solution used in this work contained 10% Pd with the density of 1.118 g/ml, the weight of the $Pd(NO_3)_2$solution needed to obtain the expected Pdwas thus;

$$\text{weight(g)of } Pd(NO_3)_2 = 10 \times A$$

From the density of 1.118 g/ml, volume of$Pd(NO_3)_2$ was

$$\text{volume (ml)} = \frac{\text{weight (g)of } Pd(NO_3)_2}{1.118\text{ g/ml}}$$

For example, to prepare 1%Pd-TUD-1, the weight (X) of Pd used was:

$$1\% = \frac{X}{X + 0.3\text{ g}} \times 100\%$$

$$\text{weight } (X) \text{ of Pd for 1\%Pd-TUD-1 } = \frac{0.3 \times 0.01}{1 - 0.01}\text{g} = 3.03 \times 10^{-3}\text{g}$$

Thus, we need to prepare Pd from the $Pd(NO_3)_2$ solution with the weight of 3.03×10^{-2}g which is equivalent to 0.0271 ml.The Table A1 summarizes the amount of $Pd(NO_3)_2$ solutionneeded for synthesizing various Pd-TUD-1 in this work.

Table A1 Amount of $Pd(NO_3)_2$ solution needed for synthesizing various Pd-TUD-1

Samples	Weight (g) of $Pd(NO_3)_2$	Volume (ml) of $Pd(NO_3)_2$
1% Pd-TUD-1	3.03×10^{-2}	0.0271
2% Pd-TUD-1	6.12×10^{-2}	0.0547
3% Pd-TUD-1	9.28×10^{-2}	0.0830
4% Pd-TUD-1	1.25×10^{-1}	0.1118
5% Pd-TUD1	1.58×10^{-1}	0.1413

Appendix B Temperature-Programmed Reduction (TPR)

Table B1 shows the condition for pretreatment process used in this work.

Table B1 Conditions for the pretreatment process prior to TPR analysis

With Gas	Flow [ccm/min]	Start at T [°C]	Ramp [°/min]	Stop at T [°C]	Hold for [min]
Nitrogen	20	Off			5
Nitrogen	20	0	10	120	30
End Pretreatment with Oven at 30°C					

Tables B2, B3, and B4 show the results from TPR analysis of 1, 3, and 5% Pd-TUD-1, respectively. These tables also indicate the times of the starting, stopping, and maximum points, temperature (°C), and the integral area (mVs) of each peak.

Table B2 The results from TPR analysis of 1% TUD-1

Peak #	Start [min]	Stop [min]	Maximum [min]	T[°C]	Integral [mVs]	[%]	[μmol/g]
1	7.1	17.6333	9.8167	127	14909.54	100	0

Table B3 The results from TPR analysis of 3% TUD-1

Peak #	Start [min]	Stop [min]	Maximum [min]	T[°C]	Integral[mVs]	[%]	[µmol/g]
1	5.75	10.3333	7.0667	100	846.5	13.43	0
2	7.4167	16.4833	10.7333	137	5455.76	86.57	0

Table B4 The results from TPR analysis of 5% TUD-1

Peak #	Start [min]	Stop [min]	Maximum [min]	T[°C]	Integral[mVs]	[%]	[µmol/g]
1	5.8	10.467	6.8167	99	1142.8	6.49	0
2	7.5833	16.35	11.083	141	16475.33	93.51	0

Appendix C Catalytic activity results from GC-MS

Table C1 illustrates %conversion, average % conversion, and SD of products resulting from GC-MS analysis in Suzuki reaction using 1, 3, and 5% Pd-TUD-1 catalysts. Figure C1 shows a plot of percent conversion versus reaction time.

Table C1 GC-MS results of TUD-1 and Pd-TUD-1

Sample	Batch	Time (min)	% Conversion	Average of % Conversion	% yield			
					major product	Av.	byproduct	Av.
Unloaded TUD-1	1	10	-	-	-	-	-	-
	2	20	-	-	-	-	-	-
	3	30	-	-	-	-	-	-
1% Pd-TUD-1	1	10	-	-	-	-	-	-
	2	20	-	-	-	-	-	-
	3	30	-	-	-	-	-	-
3% Pd-TUD-1	1	10	19		19		-	-
	2		12	17±5	12	17±5	-	-
	3		21		21		-	-
	1	20	1		1		-	-
	2		1	1±0.5	1	1±0.5	-	-
	3		2		2		-	-

Sample	Batch	Time (min)	% Conversion	Average of % Conversion	% yield			
					major product	Av.	byproduct	Av.
3% Pd-TUD-1	1	30	2	2±1	1	1	1	1±0.6
	2		1		1		1	
	3		3		1		2	
5% Pd-TUD-1	1	10	20	20±2	20	20±2	-	-
	2		21		21		-	-
	3		18		18		-	-
	1	20	6	6±1	6	6±1	-	-
	2		4		4		-	-
	3		6		6		-	-
	1	30	12	14±2	5	4±0.6	7	10±3
	2		17		4		13	
	3		14		4		10	

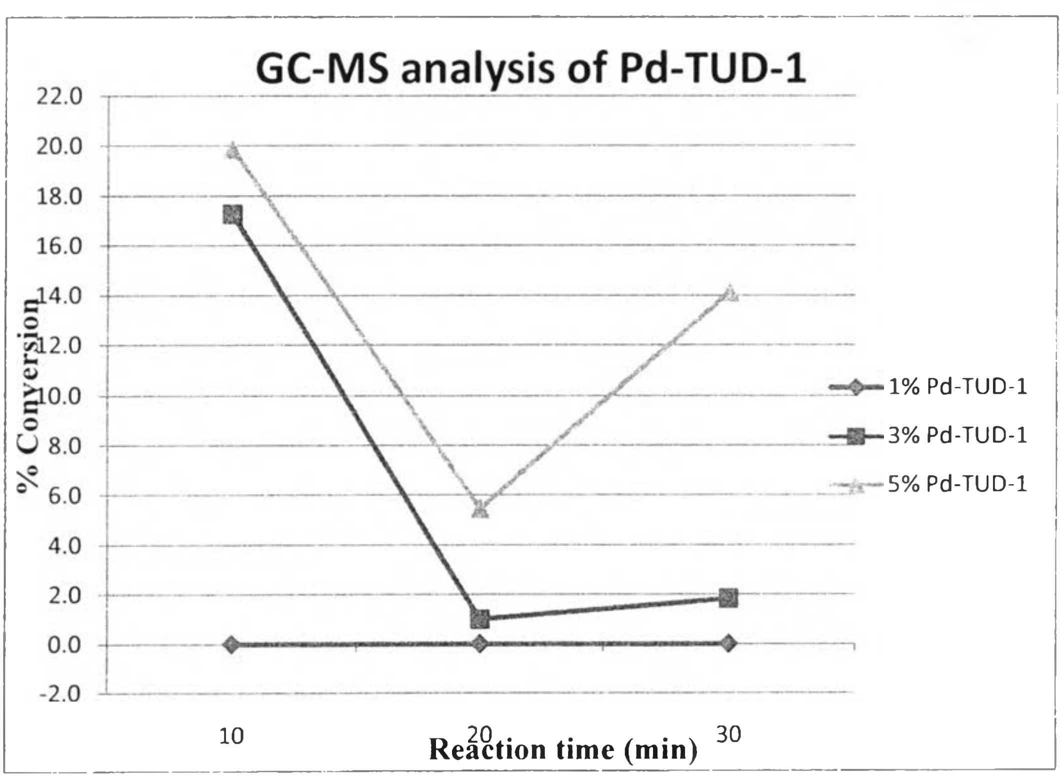

Figure C1 Catalytic activities of 1, 3, and 5% Pd-TUD-1.

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Presentations:

1. Hopetrungruang, S.; Chaisuwan, T.; Luengnaruemitchai, A.; and Wongkasemjit, S. (2013, March 3-7) Synthesis of Pd-TUD-1 via impregnation technique and its application. Paper presented at Third International Conference on Multifunctional, Hybrid and Nanomaterials 2013, Sorrento, Italy.
2. Hopetrungruang, S.; Chaisuwan, T.; Luengnaruemitchai, A.; and Wongkasemjit, S. (2013, April 23) Synthesis of Pd-TUD-1 via impregnation technique and its application. Proceedings of the 4th Research Symposium on Petrochemical and Materials Technology and the 19th PPC Symposium on Petroleum, Petrochemicals, and Polymers, Bangkok, Thailand.