

Electronic Supplementary Material (ESI) for RSC Advances.

Electronic Supplementary Information (ESI)

A Copper based Catalyst for Poly-Urethane synthesis from Discarded Motherboard

Soumitra Barman, Bibudha Parasar, Pradip Kundu, and Soumyajit Roy^{*a,b}

Eco-Friendly Applied Materials Laboratory, Department of Chemical
Sciences, New Campus, IISER-Kolkata, India; School of Chemistry and Materials
Engineering, Changshu Institute of Technology, Changshu, P. R. China

E-mail: s.roy@iiserkol.ac.in

1 Calculation of Acid value and Hydroxyl value

1.1 Acid values of the bamboo polyol

Acid value of the prepared bamboo polyol is calculated according to a previously reported procedure and is subsequently used for determination of hydroxyl value.¹ 1 g bamboo-polyol sample is first mixed with 20 mL dioxane-water solution (4/1, v/v). The resulting mixture is titrated against 1 M NaOH till the pH reaches 8.3 to indicate the end-point.

1.2 Calculation of Acid value of the bamboo polyol

$$\text{Acid Value} = (V_{\text{NaOH}(\text{sample})} - V_{\text{NaOH}(\text{blank})}) \times N_{\text{NaOH}} \times 56.1 / W_{\text{sample}}$$

Where,

$V_{\text{NaOH}(\text{sample})}$ = volume of NaOH standard solution consumed in sample titration (mL)

$V_{\text{NaOH}(\text{blank})}$ = volume of NaOH standard solution consumed in blank titration (mL)

W_{sample} = sample weight (g)

N_{NaOH} = equivalent concentration of NaOH standard solution (M) = 1(M)

Measurement result

n	Sample(g)	$[V_{NaOH(sample)} - V_{NaOH(blank)}]$ (ml)	Acid Value(mg/g)
1.	1.0001	0.4	16.83
2.	1.0003	0.3	
3.	1.0004	0.3	

1.3 Hydroxyl value of bamboo polyol

Phthalic anhydride solution is prepared by dissolving 150 g phthalic anhydride in a 1 L mixture of dioxane and pyridine (9/1, v/v). 10 mL of the phthalic anhydride solution is mixed with 1 g of polyol sample and the final mixture is added into a 150 mL beaker covered with aluminum foil. The beaker is put into a boiling water bath for 20 min. After cooling it down, 20 mL of dioxane-water solution (4/1, v/v) and 5 mL of water are added to the beaker. The resulting mixture is titrated against 1 M NaOH till the pH reaches pH 8.3 to indicate the end-point. Blank titration is conducted using the same procedure.

1.4 Calculation of Hydroxyl value of the bamboo polyol

$$\text{Hydroxyl value} = (V_{NaOH(blank)} - V_{NaOH(sample)}) \times N_{NaOH} \times \frac{56.1}{W_{sample}} + \text{Acid value}$$

Where,

$V_{NaOH(sample)}$ = volume of NaOH standard solution consumed in sample titration (mL)

$V_{NaOH(blank)}$ = volume of NaOH standard solution consumed in blank titration (mL)

W_{sample} = sample weight (g)

N_{NaOH} = equivalent concentration of NaOH standard solution (M).

Measurement result

n	Sample(g)	[V _{NaOH(blank)} -V _{NaOH(NaOH)}] (ml)	Hydroxyl Value(mg/g)
1.	1.0000	3.0	185.13
2.	1.0003	3.0	
3.	1.0002	3.0	

2 TGA data of DBSNa and Cu-based catalyst

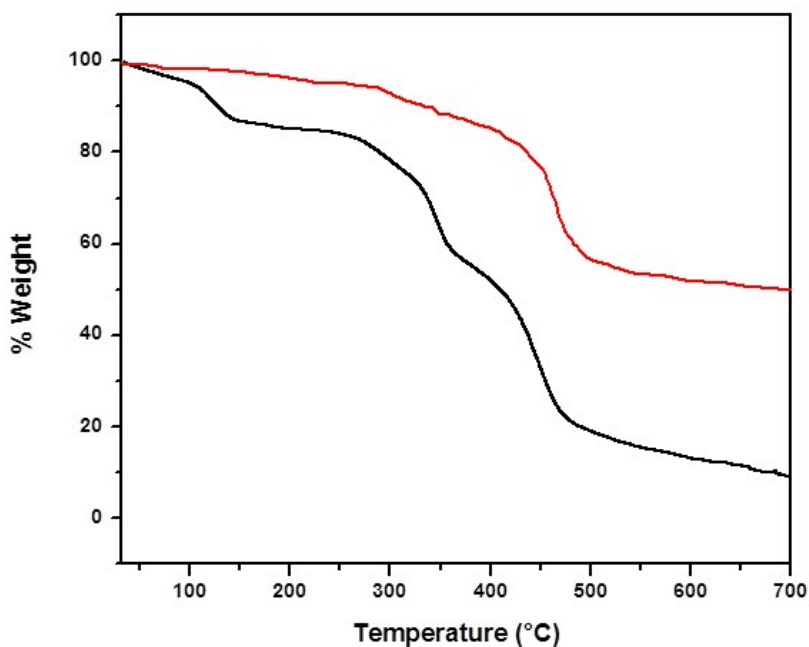


Fig. S1 TGA measurements of DBSNa (in red) and Cu-based catalyst (in black).

3 Model reactions with IPDI and Benzyl alcohol

3.1 Preparation of Benzyl((5-(((benzyloxy)carbonyl)amino)-1,3,3-trimethylcyclohexyl)methyl)carbamate

In a 50 ml round bottom flask, IPDI (1 mmol) and Benzyl alcohol (2.2 mmol) was taken and dissolved in 10 ml acetonitrile. Then Cu-based catalyst (5 mol%) was added to the solution

and heated at 60^o C. The progress of the reaction was controlled by TLC. Product was separated by column chromatography with 5% EtOAc in hexane as eluent.

¹H NMR (400 MHz, CDCl₃): δ = 7.53-7.49 (m, 1H), 7.3 (m, 10H), 7.21-7.18 (m, 1H), 4.61 (s, 4H), 4.14-4.10 (m, 1H), 3.18 (m, 2H), 1.08-1.04 (m, 6H), 1.02-0.89 (m, 9H).

¹³C NMR (100 MHz, CDCl₃): δ = 156.8, 155.6, 140.8, 128.3, 127.9, 126.7, 64.7, 54.6, 46.7, 44.4, 36.1, 34.8, 31.5, 27.3, 22.9, 13.9

Table S1

IPDI	Benzyl Alcohol	Catalyst used	Amount of catalyst used	Reaction has occurred (Yes/No)	Time (h)	Yield (%)
1mmol	2.2 mmol	DBSNa	5mol%	No	NA	NA
1mmol	2.2 mmol	Cu Powder	5mol%	Yes	18	63
1mmol	2.2mmol	Cu-based Catalyst	5mol%	Yes	6	85

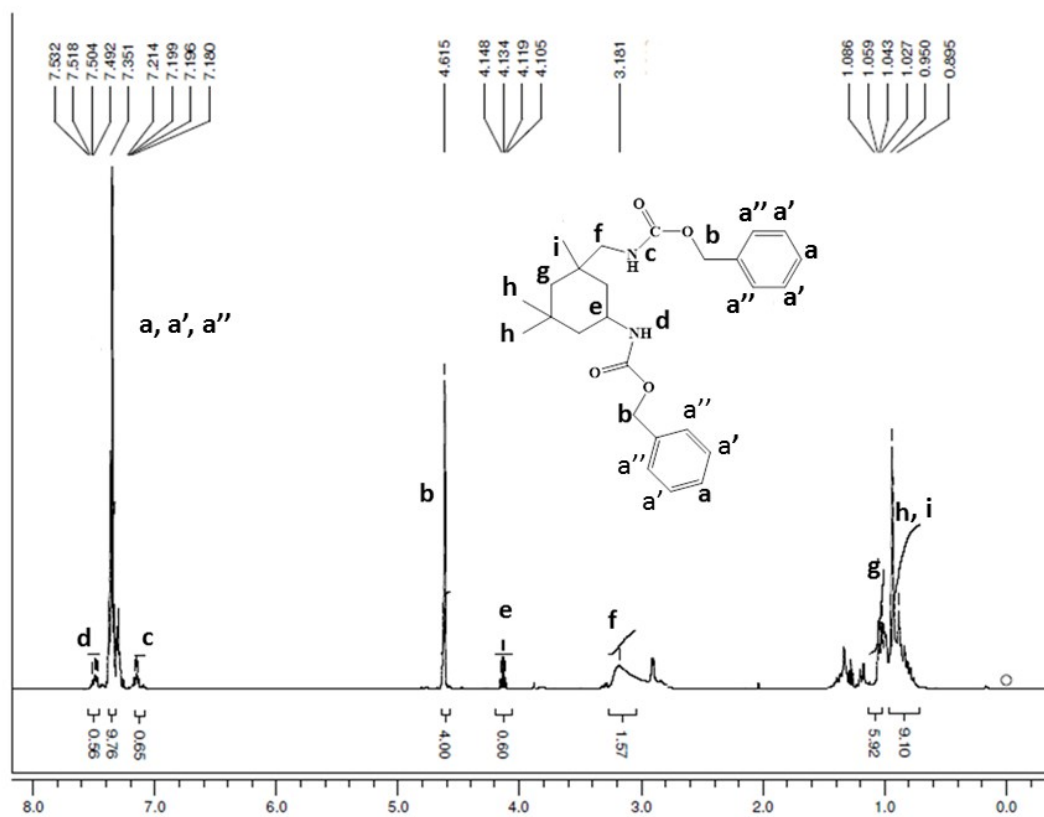


Fig. S2 ^1H NMR Spectra (CDCl_3 , 25°C) of Benzyl((5-(((benzyloxy)carbonyl)amino)-1,3-trimethylcyclohexyl)methyl)carbamate.

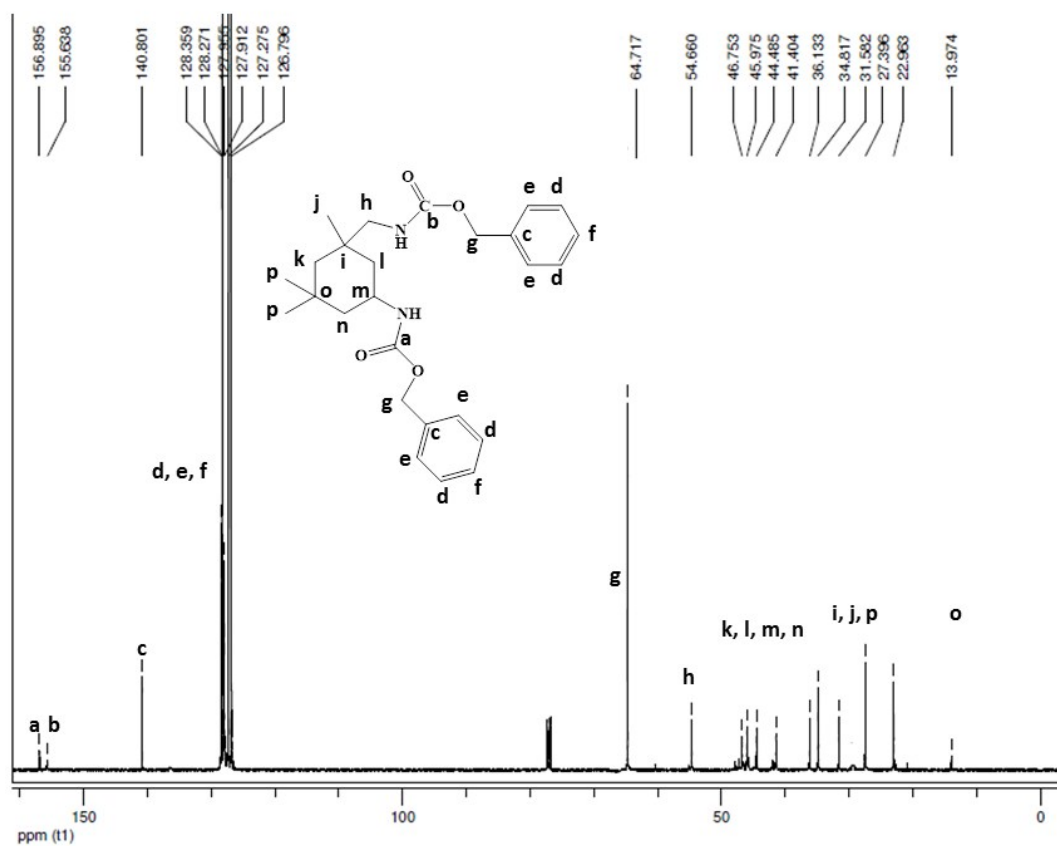


Fig. S3 ^{13}C NMR spectra (CDCl_3 , 25°C) of Benzyl((5-(((benzyloxy)carbonyl)amino)-1,3,3-trimethylcyclohexyl)methyl)carbamate.

References

1. L. L. Gao, Y. H. Liu, H. Lei, H. Peng and R. Ruan, *J. Appl. Polym. Sci.*, 2010, **116**, 1694–1699.