Electronic supplementary information

Facile single step synthesis of acid functionalized nano porous carbon composite as efficient catalyst for tertiary butylation of phenol

Devaki Nandan[ab] and Nagabhatla Viswanadham[ab]*

[a]*Academy of Scientific and Innovative Research (AcSIR) at CSIR-Indian Institute of Petroleum Dehradun-248005 (Uttarakhand), India*

[b]*Refining Technology Division, Indian Institute of Petroleum, Council of Scientific and Industrial Research, Dehradun-248005, Indi,. Fax: 91-135-2525702, Tel: 91-135-2525856, E-mail: nvish@iip.res.in*

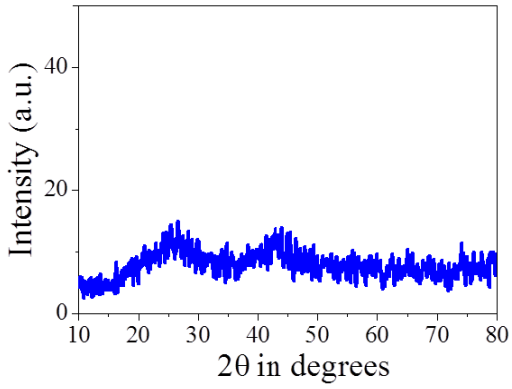
1. Experimental Details:

Powder X-ray diffraction patterns of the samples were recorded on a Regaku Dmax III B equipped with rotating anode and CuKα radiations. SEM images were recorded for obtaining particle morphology on Quanta 200f instrument, Netherland. The IR spectra of the both samples were recorded on Thermonicolate 8700 instrument, Thermoscientific Corporation, USA. The reaction product is analyzed using GC equipped with the DBwax column and FID detector. The catalyst performance towards alkylation of Phenol was done in present work. In a typical reaction procedure, 1 mol of phenol was added to a 2.5 mol of tertiary butyl alcohol and 5 mol% catalyst and the whole mixture was transferred to a Parr reactor autoclave of 25 ml volume capacity, sealed tightly and pressurised by N_2 up to 1 bar. The reaction was conducted at 403 K for 5h and the product obtained at the end of the run was filtered and analysed by GC.

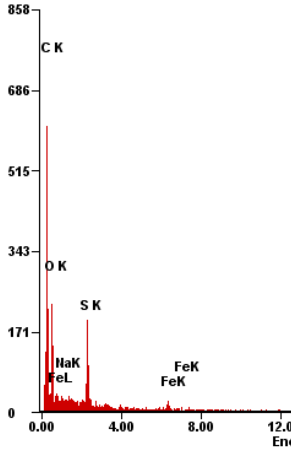
Supporting Table 1: Textural properties of NPC

Sample	SA_{BET} m^2g^{-1} [a]	V_{tot} cm^3g^{-1} [b]	C%[c]	S%[c]	Acid density due to $-SO_3H$ (mmol/g)	Total Acidity[d]
NPC	7.6	0.0252	59	4.57	1.43	4.03
[a]BET surface area. [b]total pore volume taken from the volume of N_2 adsorbed at P/P_0 = 0.995. [e]micropore volume calculated from t-plot. [C]Determined by CHNS elemental analysis. [d]Determined by acid base titration						

Supporting Fig. S1 XRD pattern of material



Supporting Fig. S2 EDX spectra of material.



Catalyst	Reaction temperature K	Phenol conversion%
HZOP-31	343	25.12[1]
H-AlMCM-48	448	59.1[2]
Sulfated titania	473	32.20[3]
Mesoporous Galosilicate	448	37.0[4]
H-Y(5.2)	403	100[5]
HPW/MCM-41	403	99[5]
H-beta(25)	403	72[5]
FeSBA-1(36)	473	78.5[6]
Ga-FSM-16	433	80.3[7]
Sulfated Fe_2O_3–TiO_2		
(Al-MCM-41 MCM-22 M-MCM-22)	418	(61.3 94.0 93.3)[8]
SCS	393	52%[9]

Supporting Table 2: Performance comparison of synthesized NPC with the reported materials for tertiary butylation of phenol

1 K. Ojha, N. C. Pradhan, A. N. Samanta, Chemical Engineering Journal, 2005, 112, 109.
2 S. E. Dapurkar, P. Selvam, Applied Catalysis A: General, 2003, 254, 239.
3 K. R. Sunajadevi and S. Sugunan, Catalysis Letters, 2005, 99, 3.
4 A. Sakthivel and P. Selvam, Catalysis Letters, 2002, 84, 1.
5 G. Kamalakar, K. Komura, and Y. Sugi, *Ind. Eng. Chem. Res.* 2006, *45,* 6118.
6 A. Vinu, T. Krithiga, V. V. Balasubramanian, A. Asthana, P. Srinivasu, T. Mori, K. Ariga, G. Ramanath, and P. G. Ganesan, *J. Phys. Chem. B*, 2006, *110,* 11924.
7 K. Bachari, R. M. Guerroudj • M. Lamouchi, Reac. Kinet. Mech. Cat., 2011, 102, 219.
8 K. Song, J. Guan, S. Wu, Y. Yang, B. Liu and Q. Kan, Catal. Lett., 2008, 126, 333.

	This work	Sulfated Fe_2O_3–TiO_2[1]	Solid sulfanilic acid[2]
Temperature	403 K	393 K[a]	393 K
Conversion in respect of phenol	85%	-	-
Conversion in respect of TBA	>97%	~40%[b]	95%
Time hours	5	9	9
Feed ratio Phenol to TBA	2	2	2

1 K. J. A. Raj, M. G. Prakash and B. Viswanathan, Catalysis Science & Technology, DOI: 10.1039/c1cy00157d.
2 F. Adam, K. M. Hello, T. H. Ali, Applied Catalysis A: General, 2011, 399, 42.

Supporting Table 3: Performance comparison of synthesized NPC material with reported materials for liquid phase tertiary butylation of phenol (similar conditions with comparable reaction temperature)

A, b conversion was given in graph. In respective paper.