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Ying Li[a, b], Deliang Duan[a], Mingzhu Wu[a, b], Junjie Li[a], Zhiying Yan[a], Wei Wang[a], Guoli Zi[a], Jiaqiang Wang[a,*]

[a] Yunnan Provincial Collaborative Innovation Center of Green Chemistry for Lignite Energy, Yunnan Province Engineering Research Center for Photocatalytic Treatment of Industrial Wastewater, The Universities' Center for Photocatalytic Treatment of Pollutants in Yunnan Province, School of Energy, School of Chemical Science & Technology, Yunnan University, Kunming 650091, P. R. China.

[b] Chemical and Materials Research Center, Chongqing Industry Polytechnic College, Chongqing 401120, P. R. China.

*Corresponding author

E-mail address: jqwang@ynu.edu.cn

Phenol and Fax: +86-871-65031567

Abstract: one-step hydroxylation of C-H (sp^2) of aromatic ring synchronized with the oxidation of C-H (sp^3) of side-chain of *p*-xylene to 2,5-dihydroxyterephathalic acid was performed using M-MCM-41 (M = Fe, Fe/Cu, Cu) as the catalysts. Cu-MCM-41 (Cu:Si = 1:100) catalyst exhibited good selectivity (73.0%) of 2,5-dihydroxyterephathalic acid and conversion (21.7%) of *p*-xylene using acetic acid and acetonitrile (V:V = 3:7) as the solvents at 80 ^{o}C for 5 h. The possible mechanism of the