# *Supplementary material*

## Synthesis and characterization of an efficient and stable Al/Fe pillared clay catalyst for the catalytic wet air oxidation of phenol

**John Moma, Jeffrey Baloyi and Thabang Ntho**

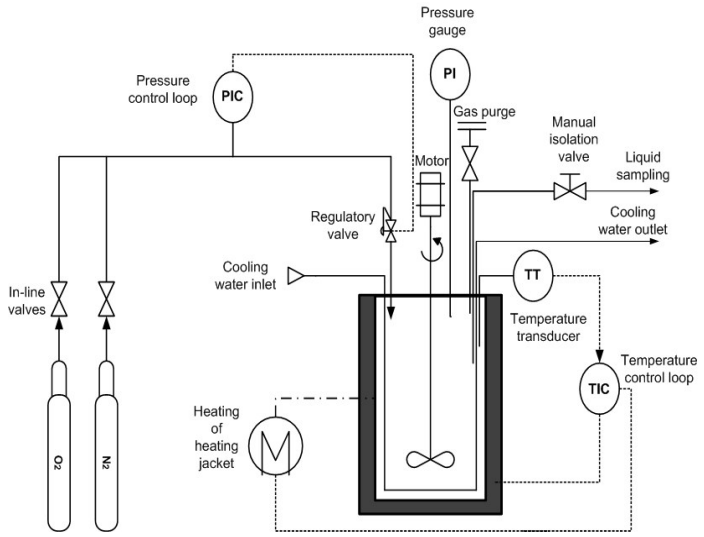



**Fig. S1:** Experimental set up for phenol oxidation semi-batch reactor system.

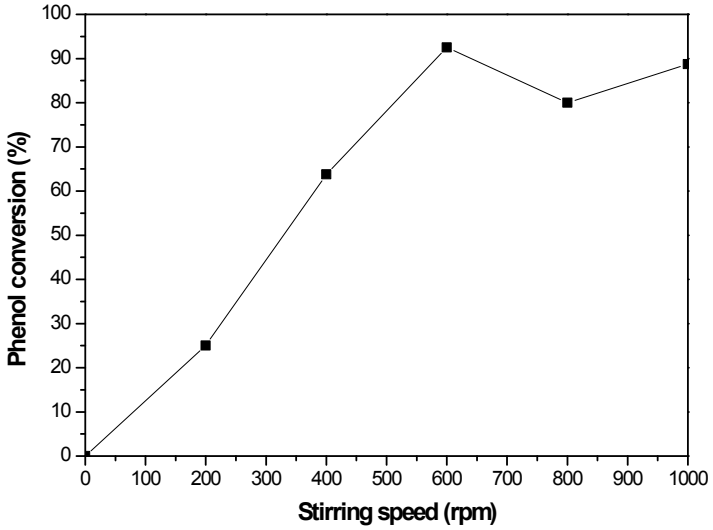



**Fig. S2:** Effect of mass transfer on the conversion of phenol in aqueous solution. (phenol conc, 1000 mg/L; volume, 200 mL; catalyst loading, 2 g/L; reaction time, 150 min; partial oxygen pressure, 10 bar; temperature, 100 °C).

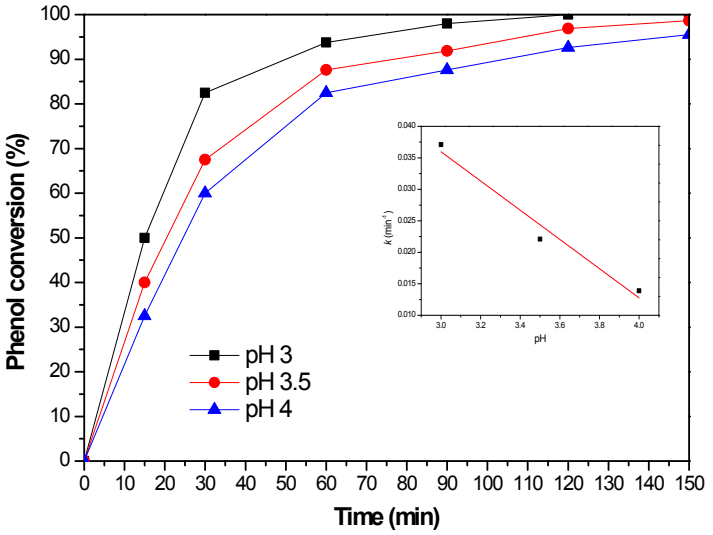



**Fig. S3:** Effect of initial pH on conversion of phenol in aqueous solution. (phenol conc, 1000 mg/L; volume, 200 mL; catalyst loading, 2 g/L; reaction time, 150 min; partial oxygen pressure, 10 bar; temperature, 100 °C)

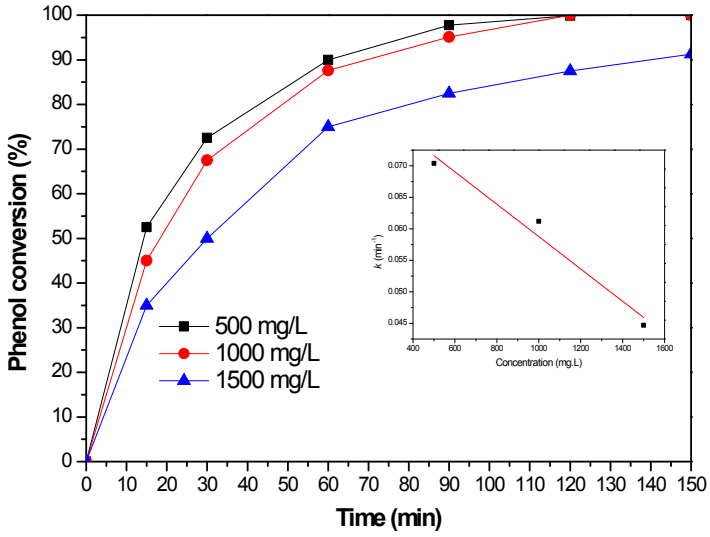



**Fig. S4:** Effect of initial concentration of phenol in aqueous solution. (volume, 200 mL; catalyst loading, 2 g/L; pH, 3.0; reaction time, 150 min; partial oxygen pressure, 10 bar; temperature 100 °C).

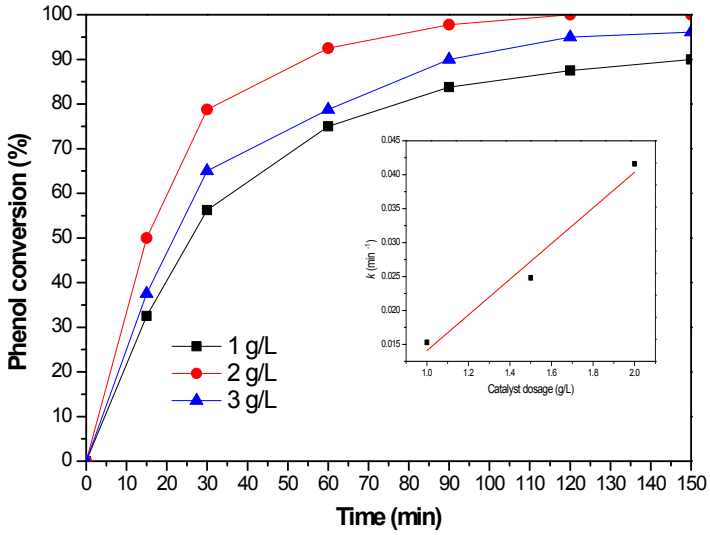



**Fig. S5:** Effect of catalyst mass on conversion of phenol in aqueous solution. (phenol concentration, 1000 mg/L; volume, 200 mL; pH, 3.0; partial oxygen pressure, 10 bars; temperature, 100 °C).

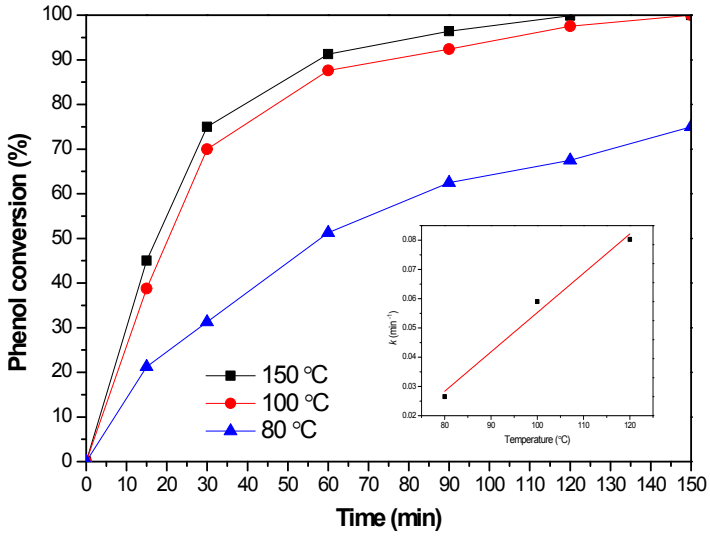



**Fig. S6:** Effect of reaction temperature on conversion of phenol in aqueous solution. (phenol concentration, 1000 mg/L; volume, 200 mL; pH, 3.0; reaction time, 180 min; partial oxygen pressure, 10 bar; catalyst loading, 2 g/L).

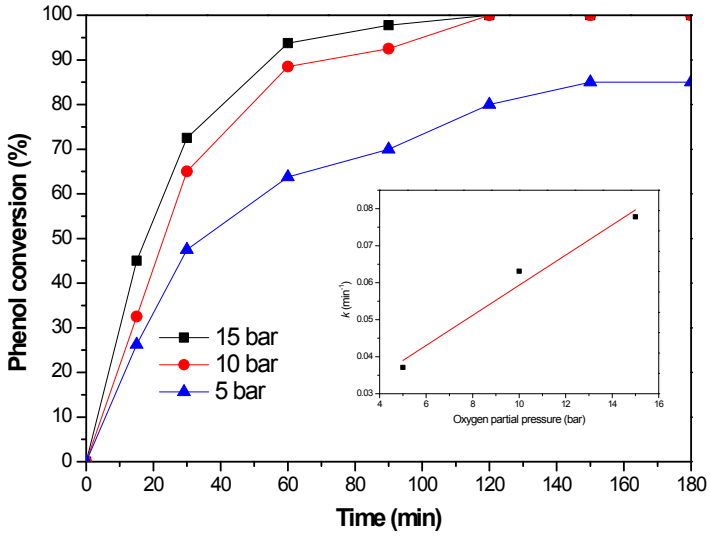



**Fig. S7:** Effect of oxygen partial pressure on conversion of phenol in aqueous solution (phenol concentration, 1000 mg/L; volume, 200 mL; pH, 3.0; reaction time, 180 min; temperature, 100 °C; catalyst loading, 2g/L).

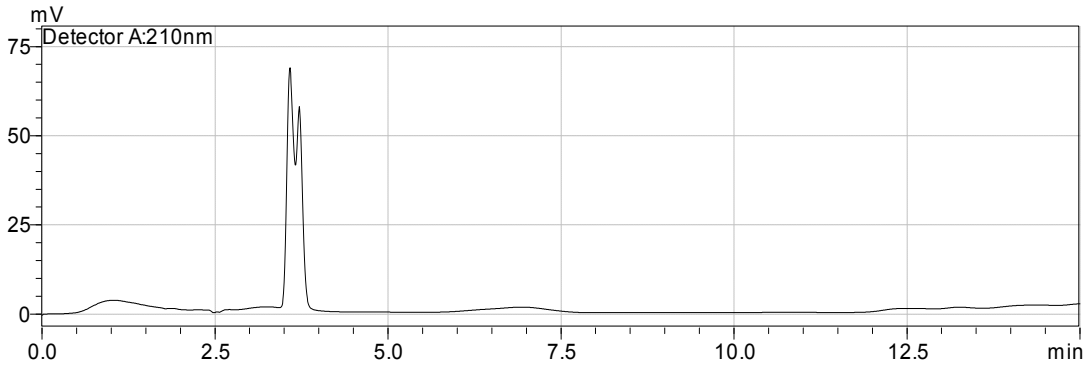



**Fig. S8:** HPLC calibration curve for phenol.

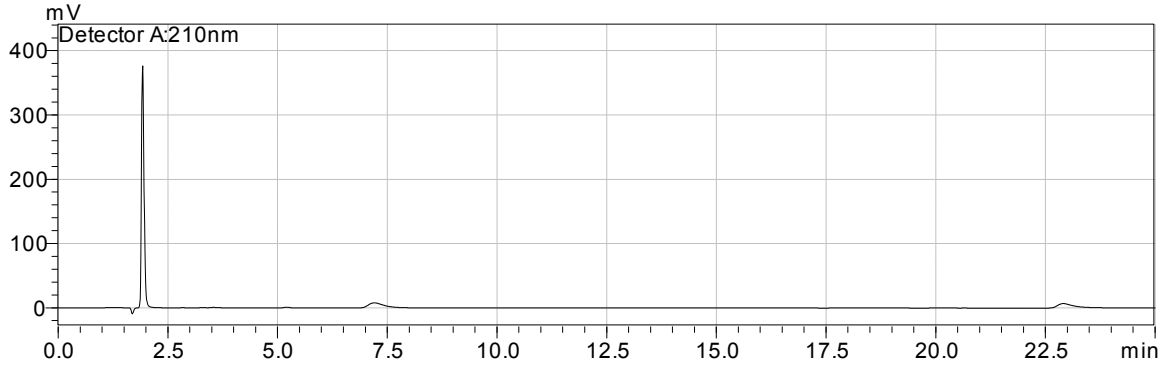



**Fig. S9:** HPLC calibration curve for acetic acid.

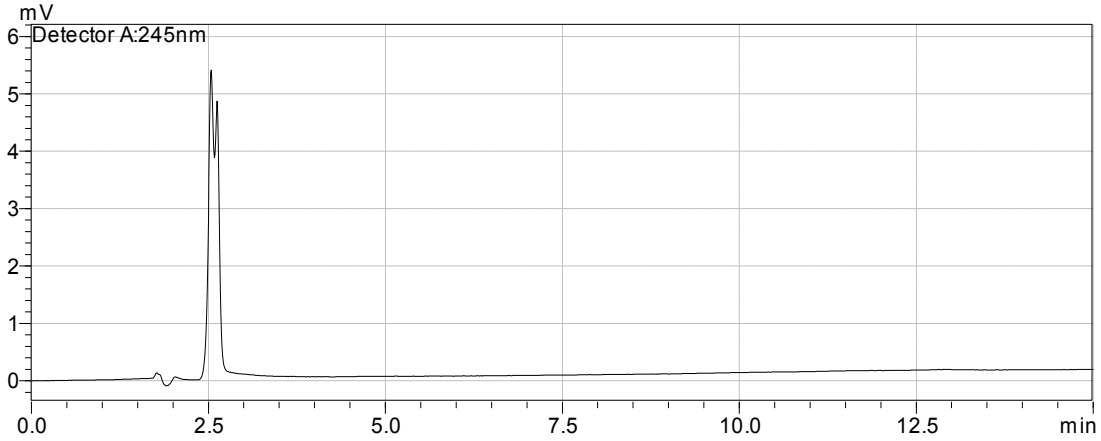



**Fig. S10:** HPLC calibration curve for formic acid.

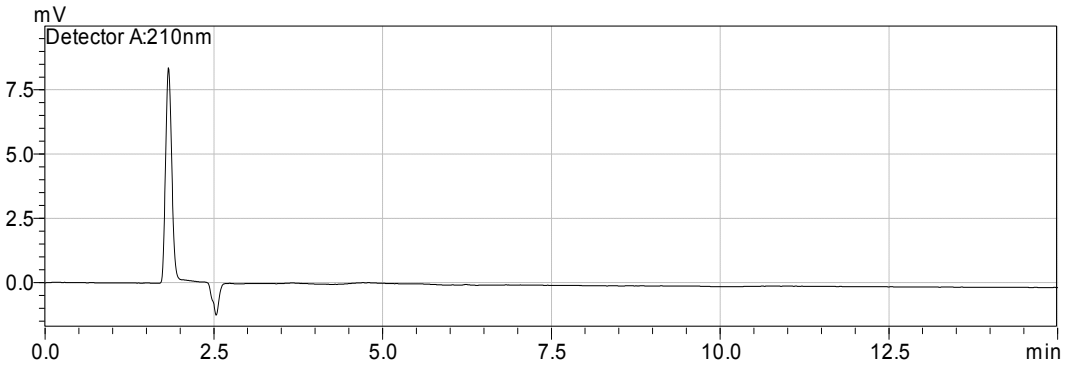



**Fig. S11:** HPLC calibration curve for maleic acid.

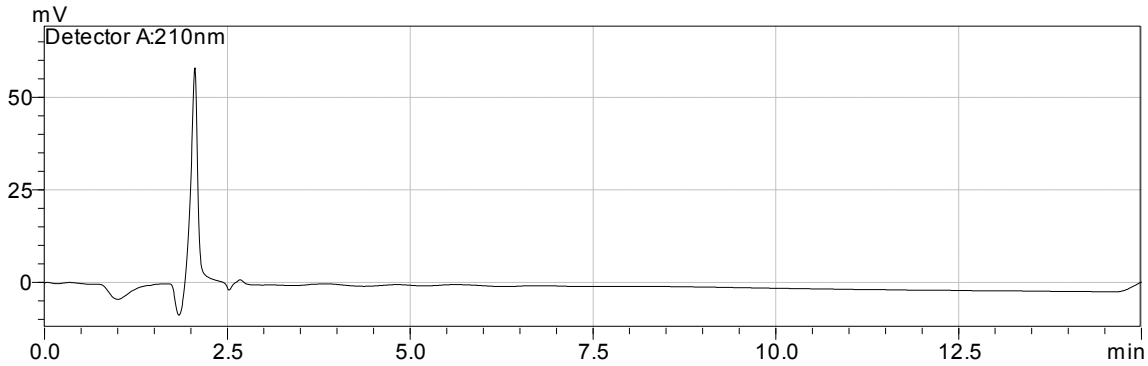



**Fig. S12:** HPLC calibration curve for hydroquinone.

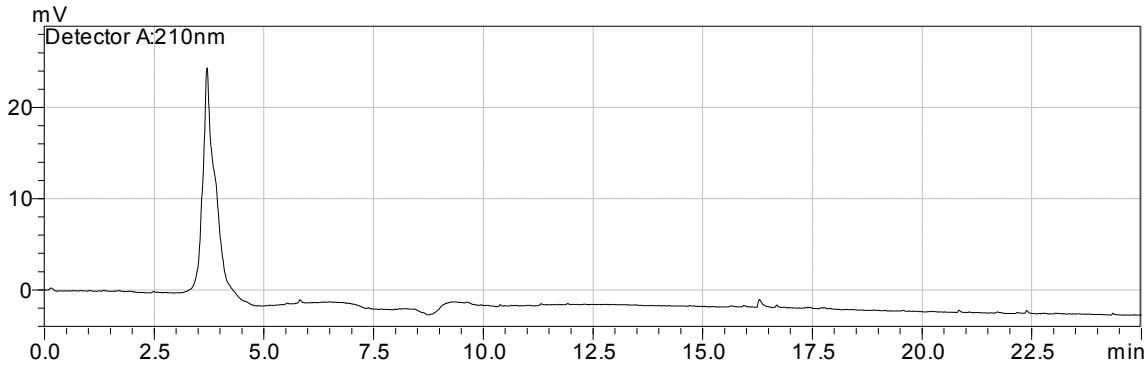



**Fig. S13:** HPLC calibration curve for pyrocatechol.

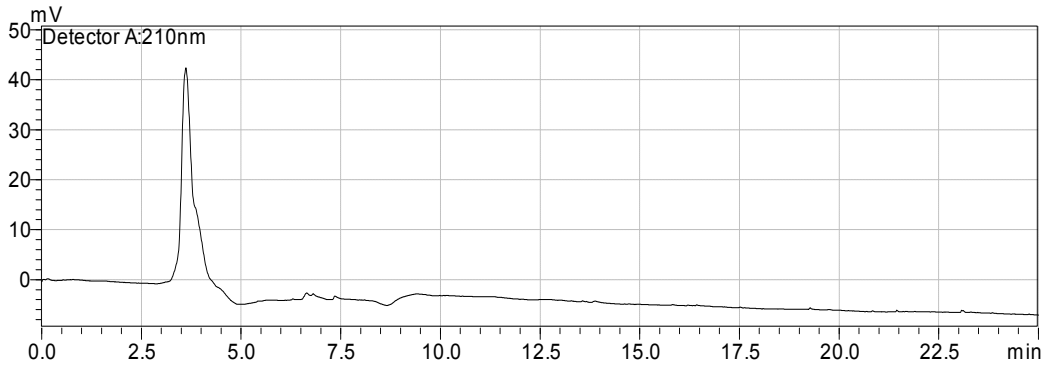



**Fig. S14:** HPLC calibration curve for benzoquinone.

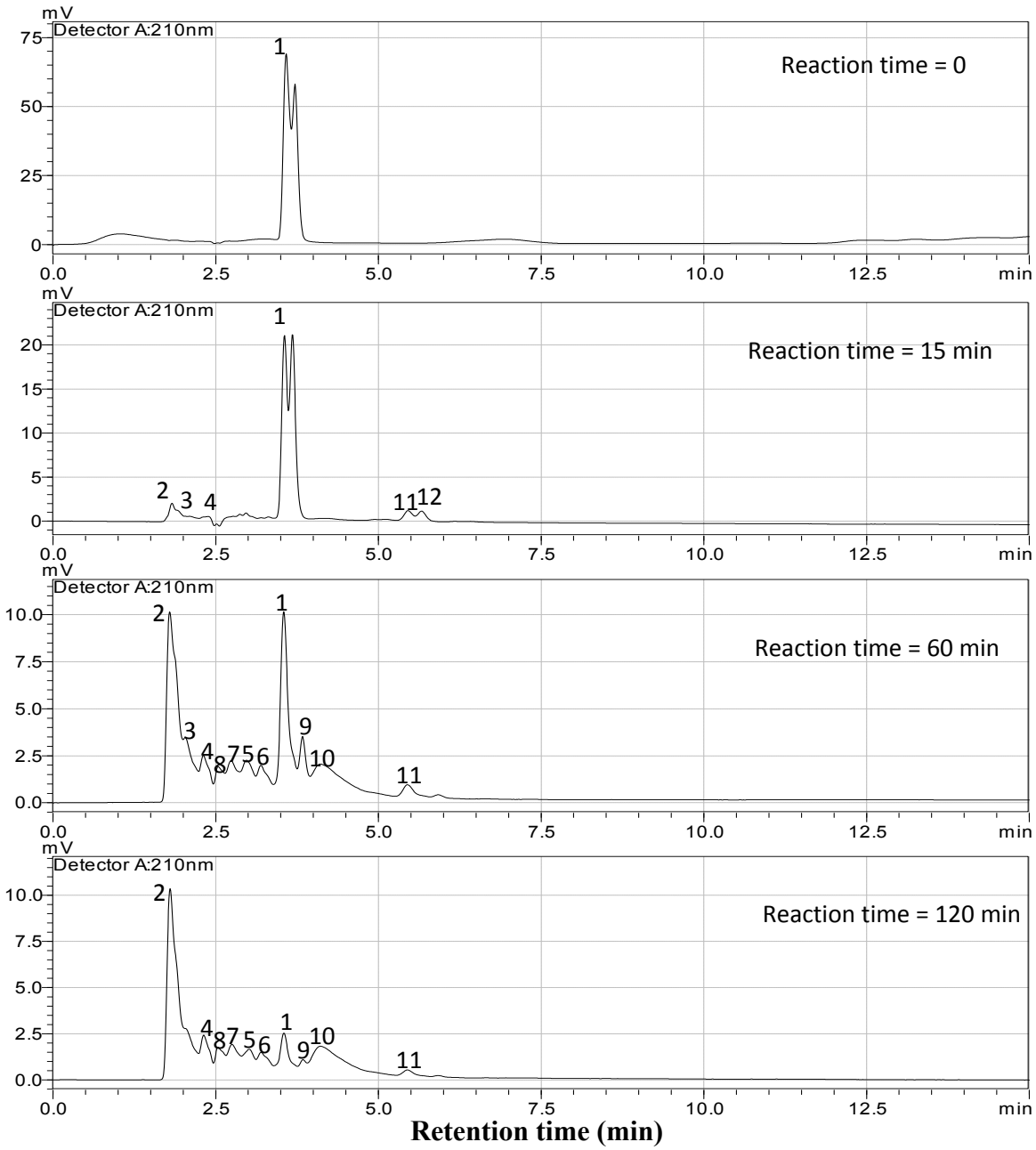
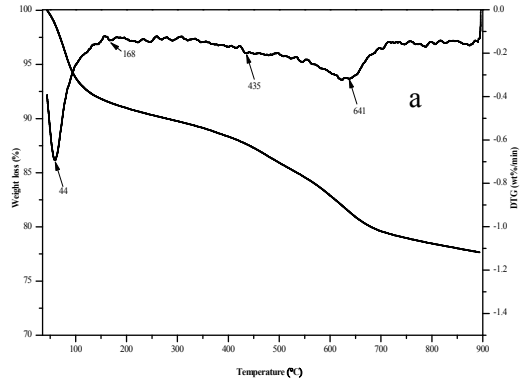
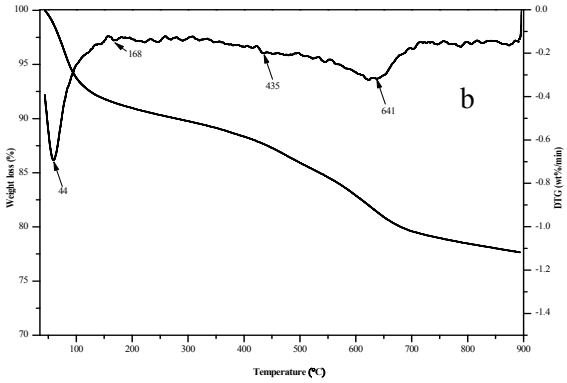
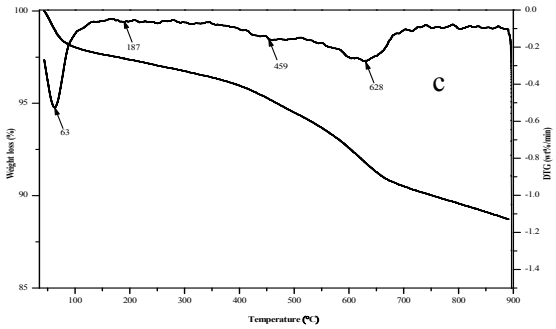
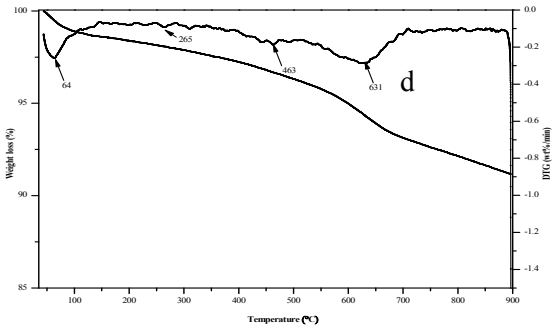


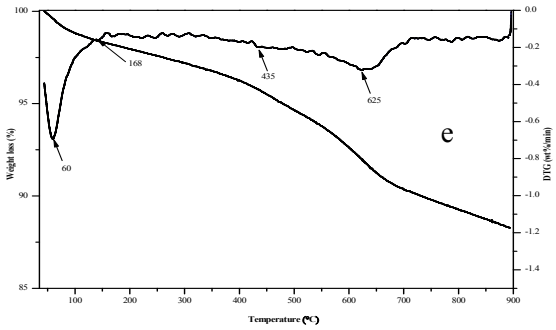

**Fig. S15:** Typical liquid chromatograms of phenol and intermediates in liquid sample. 1. Phenol; 2. Acetic acid; 3. Hydroquinone; 4. Maleic acid; 5. Benzoquinone; 6. Pyrocatechol; 7. Malonic acid; 8. Formic acid; 9. Oxalic acid.

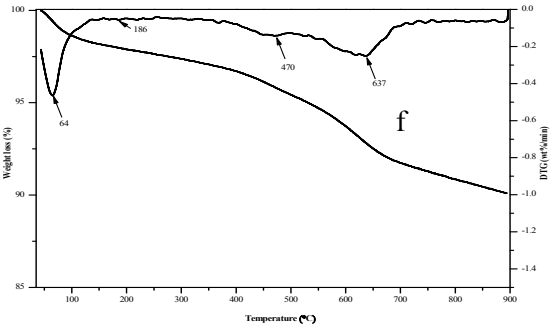

a
Weight loss (%)
DTG (wt%/min)
Temperature (℃)
44
168
435
641

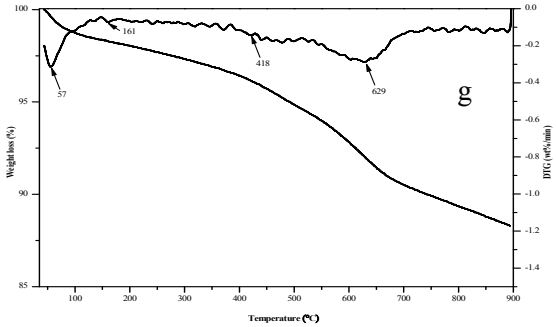

b
Weight loss (%)
DTG (wt%/min)
Temperature (℃)
44
168
435
641


c
Weight loss (%)
DTG (wt%/min)
Temperature (℃)
63
187
459
628


d
Weight loss (%)
DTG (wt%/min)
Temperature (℃)
64
265
463
631


e
Weight loss (%)
DTG (wt%/min)
Temperature (℃)
60
168
435
625


f
Weight loss (%)
DTG (wt%/min)
Temperature (℃)
64
186
470
637


g
Weight loss (%)
DTG (wt%/min)
Temperature (℃)
57
161
418
629

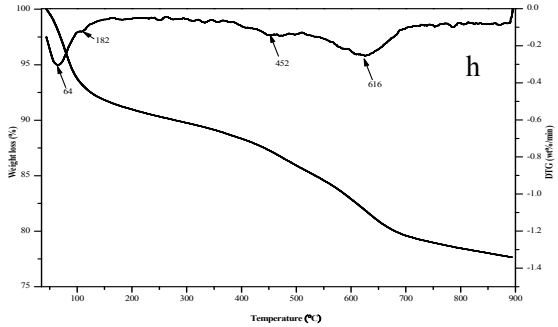

h
Weight loss (%)
DTG (wt%/min)
Temperature (℃)
64
182
452
616

**Fig. 16:** Thermogravimetric analyses (TGA) and their derivative plots (DTG) of (a) (a) natural bentonite clay, (b) Al-PILCs, (c) Fe-PILCs, (d) Al/Fe-PILCs (1:3), (e) Al/Fe-PILCs (1:1), (f) Al/Fe-PILCs (3:1) and (g) Al/Fe-PILCs (6:1).