Catalytic Liquefaction of Kraft Lignin with Solvothermal Approach

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Electronic Supplementary Information

1. External mass transfer limitations

The stirring rate affects the mass transfer of a given reaction, in our case the reaction involves two phases, the liquid and the solid catalyst. In order to properly evaluate the performance of a catalyst an efficient contact between the liquid and the catalyst must be verified. Three tests with 500, 1000 and 1500 rpm were carried out in the presence of Pt/Al_2O_3 (Figure S1). The observed small variations on the phase distribution do not show any external mass transfer limitations. The analysis of OP fraction, on the other hand, shows that at 500 rpm the amount of guaiacol produced is much higher than the reference, while the effect on the other compounds remained limited. Increasing the stirring rate seems to affect the guaiacol yield, probably due to partial degradation. Henceforth the test will be carried out at 1000 rpm, at 1500 the transfer is improved but to prevent mechanical wearing the rate was reduced. Even though with 1000 rpm the transfer at the catalyst surface is not highly efficient, we can assume its effect can still be evaluated.

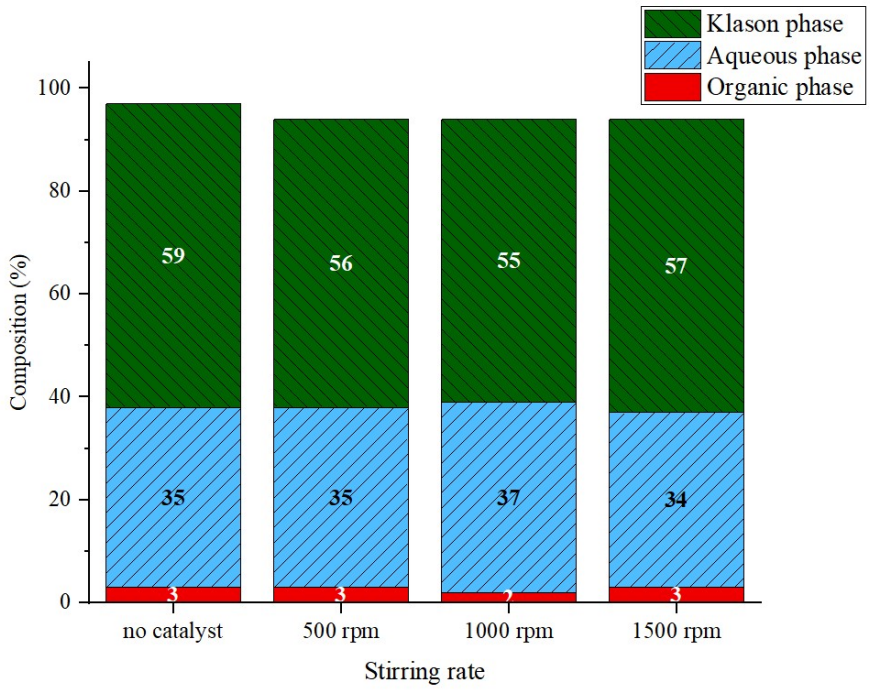

Figure S1. Phase distribution of lignin solution after treatment in the presence of Pt/Al_2O_3 with different stirring rate (225°C, 40 bar after 3 h treatment)

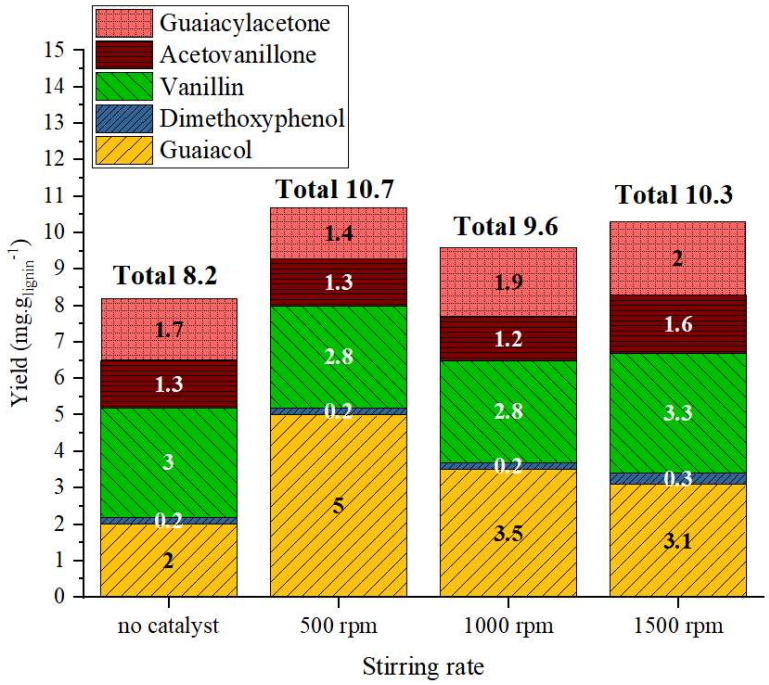

Figure S2. Yield of identified monomeric products in the presence of Pt/Al_2O_3 catalyst (225°C, 40 bar, 3 h treatment) for different stirring rates.

2. ^{1}H NMR results

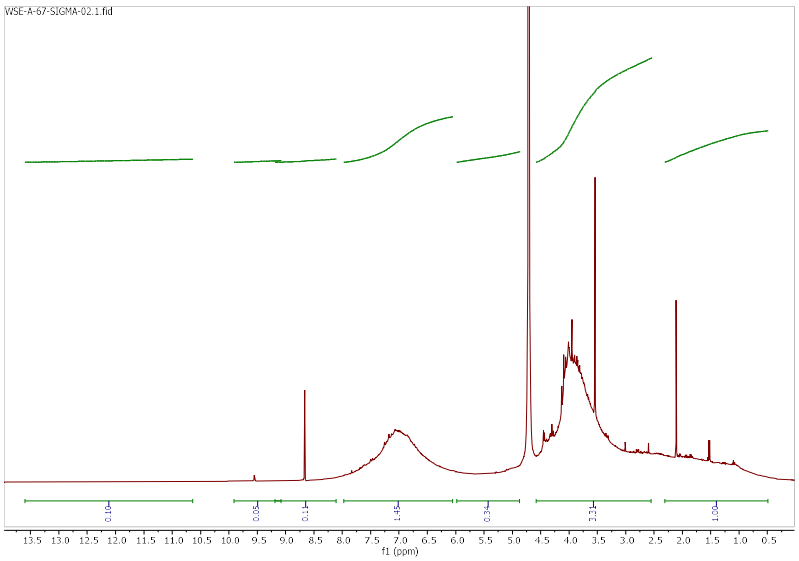

Figure S3. ^{1}H NMR spectra of initial lignin

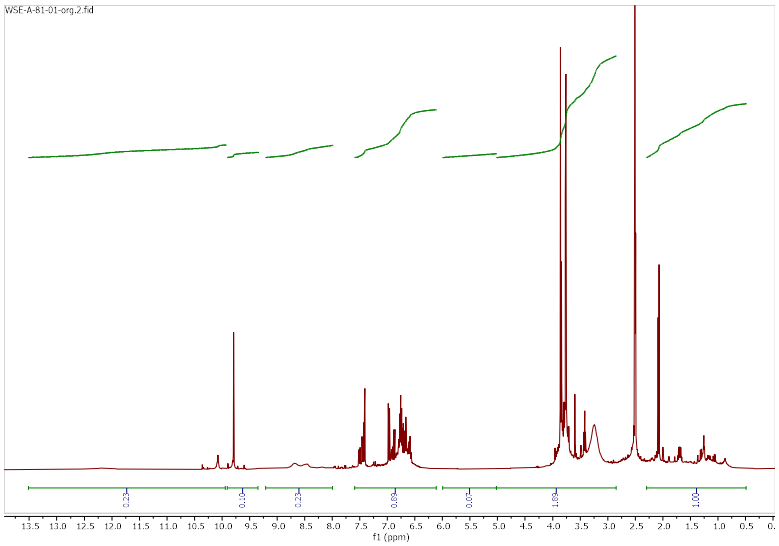

Figure S4. ^{1}H NMR spectra of OP phase obtained after reaction without catalyst in water

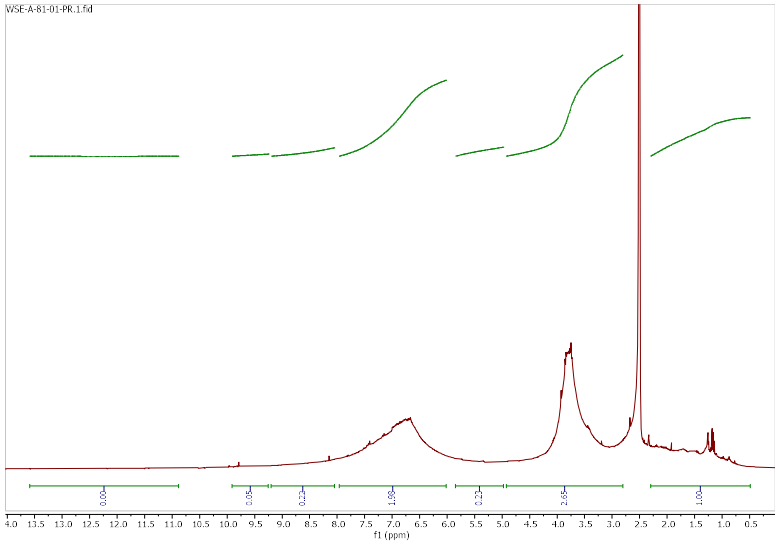

Figure S5. ^{1}H NMR spectra of KP phase obtained after reaction without catalyst in water

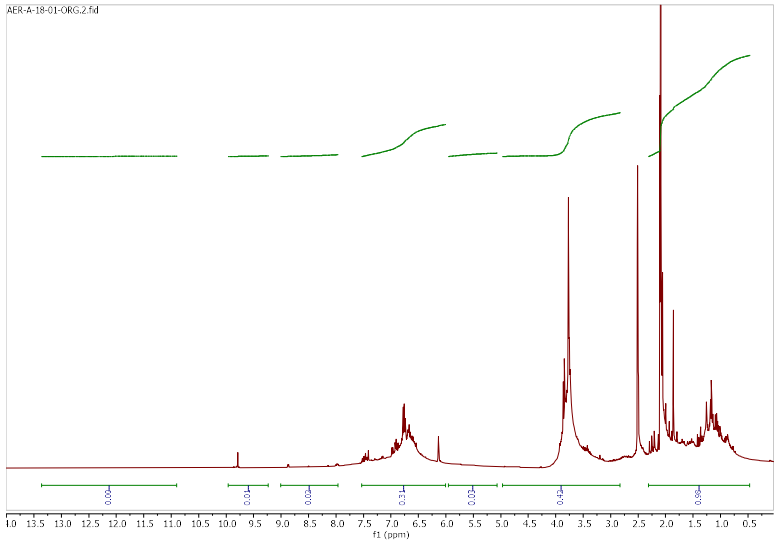

Figure S6. ^{1}H NMR spectra of OP phase obtained after reaction without catalyst in water/MeOH mixture

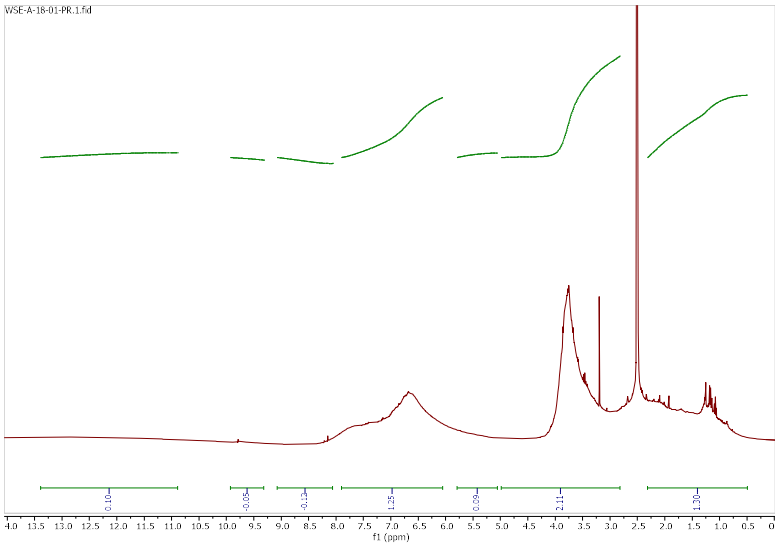

Figure S7. ^{1}H NMR spectra of KP phase obtained after reaction without catalyst in water/MeOH mixture

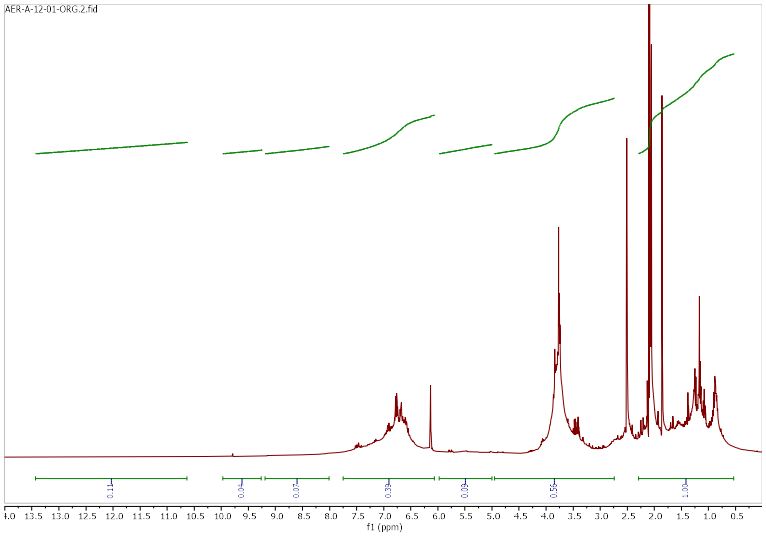

Figure S8. ^{1}H NMR spectra of OP phase obtained after reaction without catalyst in water/EtOH mixture

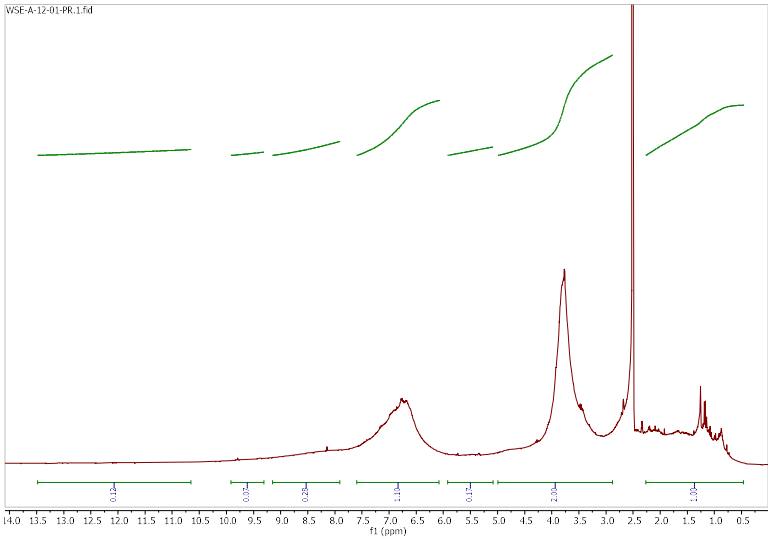

Figure S9. ^{1}H NMR spectra of KP phase obtained after reaction without catalyst in water/EtOH mixture

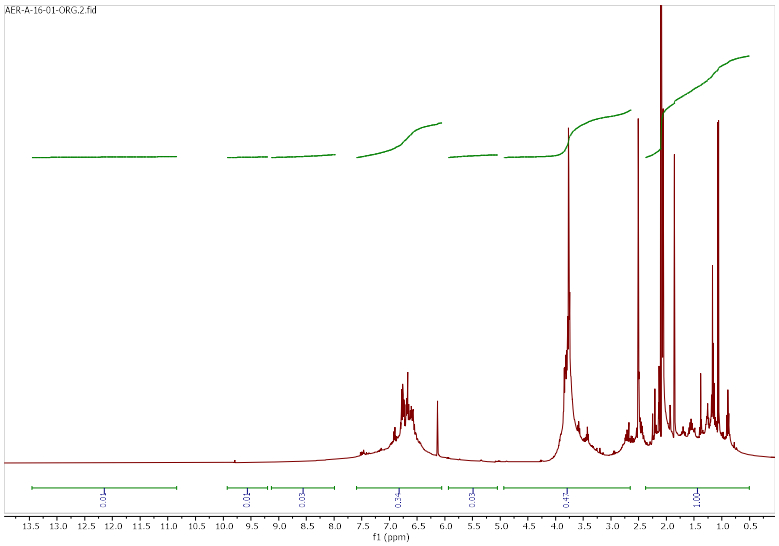

Figure S10. ^{1}H NMR spectra of OP phase obtained after reaction without catalyst in water/iPrOH mixture

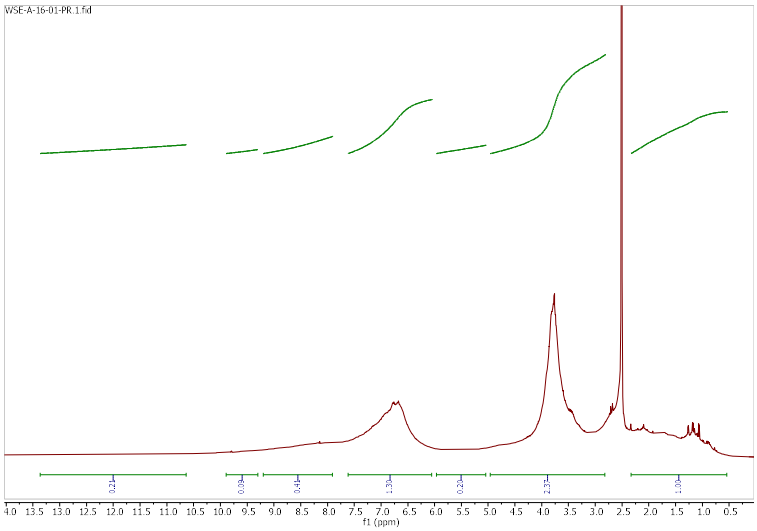

Figure S11. ^{1}H NMR spectra of KP phase obtained after reaction without catalyst in water/iPrOH mixture

3. HSQC NMR results

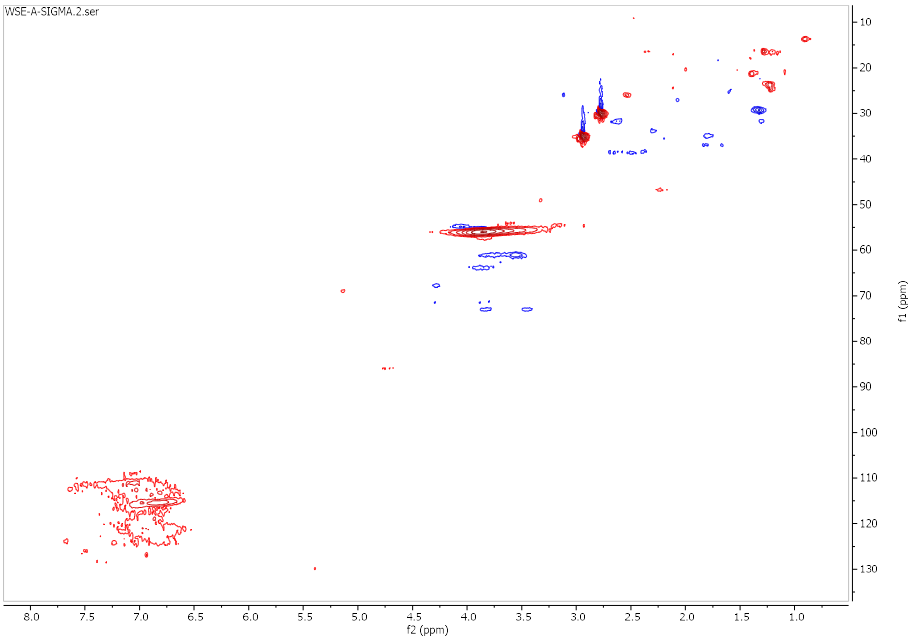

Figure S12. HSQC NMR spectra of initial lignin

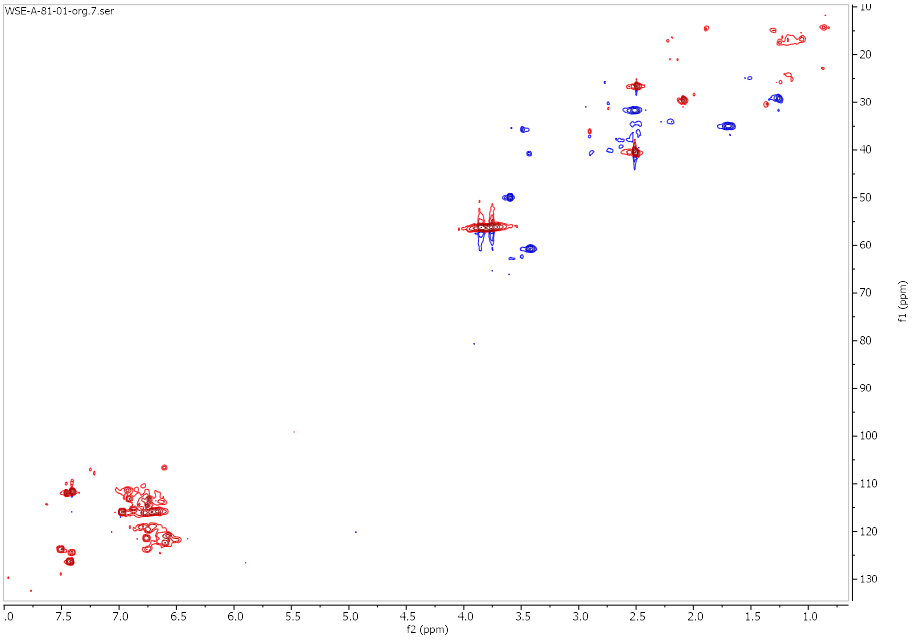

Figure S13. HSQC NMR spectra of OP phase obtained after reaction in water

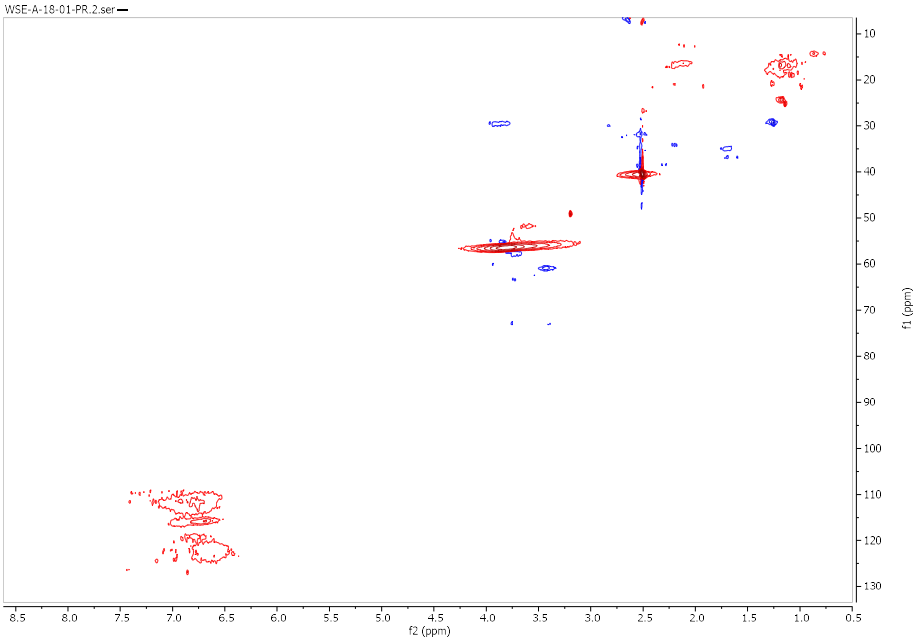

Figure S14. HSQC NMR spectra of KP phase obtained after reaction in water

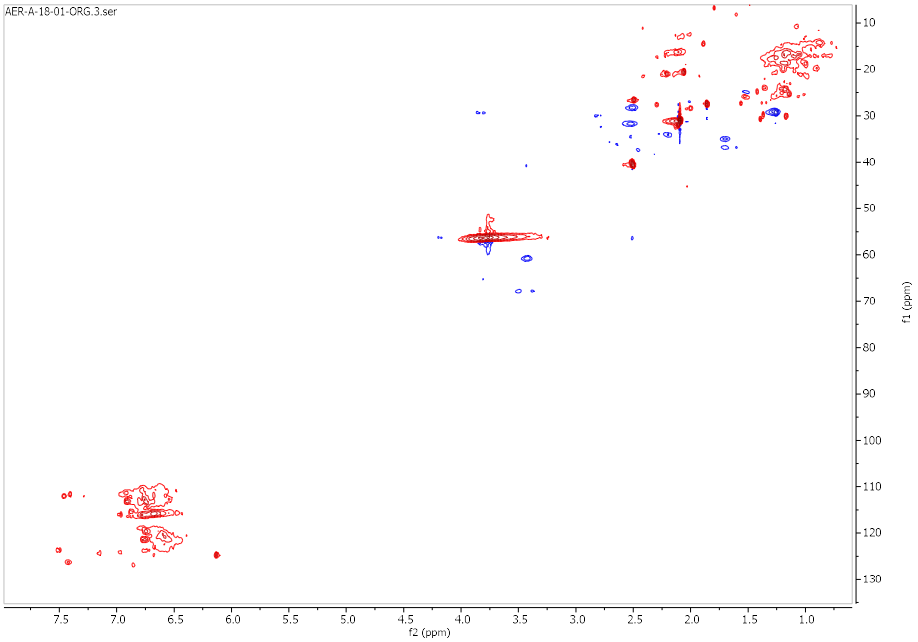

Figure S15. HSQC NMR spectra of OP phase obtained after reaction in water/MeOH mixture

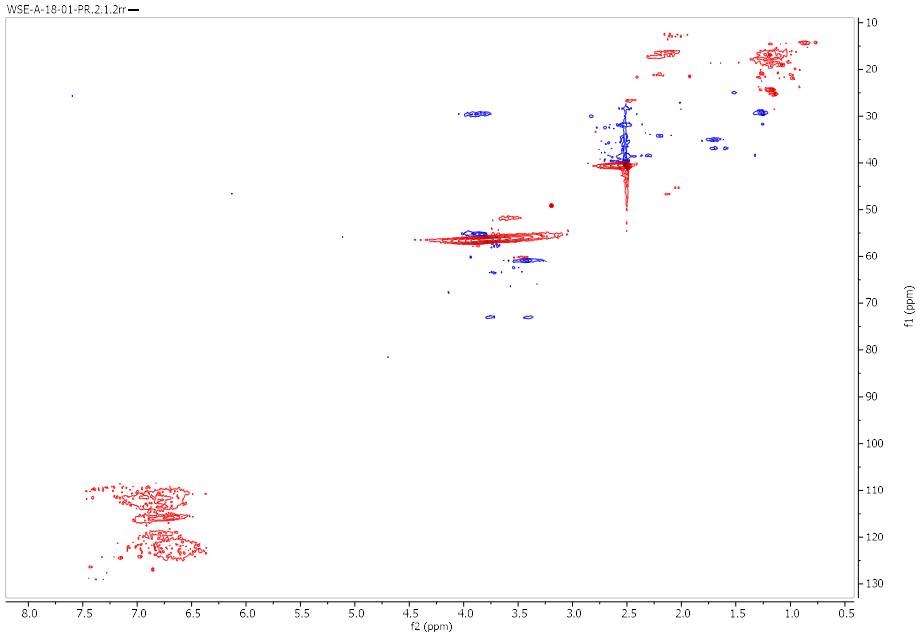

Figure S16. HSQC NMR spectra of KP phase obtained after reaction in water/MeOH mixture

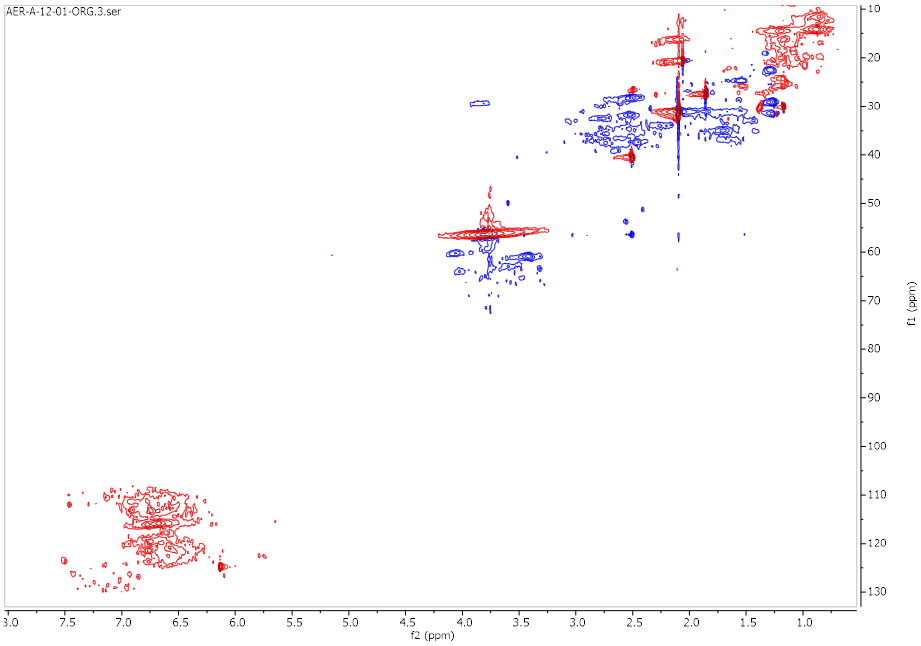

Figure S17. HSQC NMR spectra of OP phase obtained after reaction in water/EtOH mixture

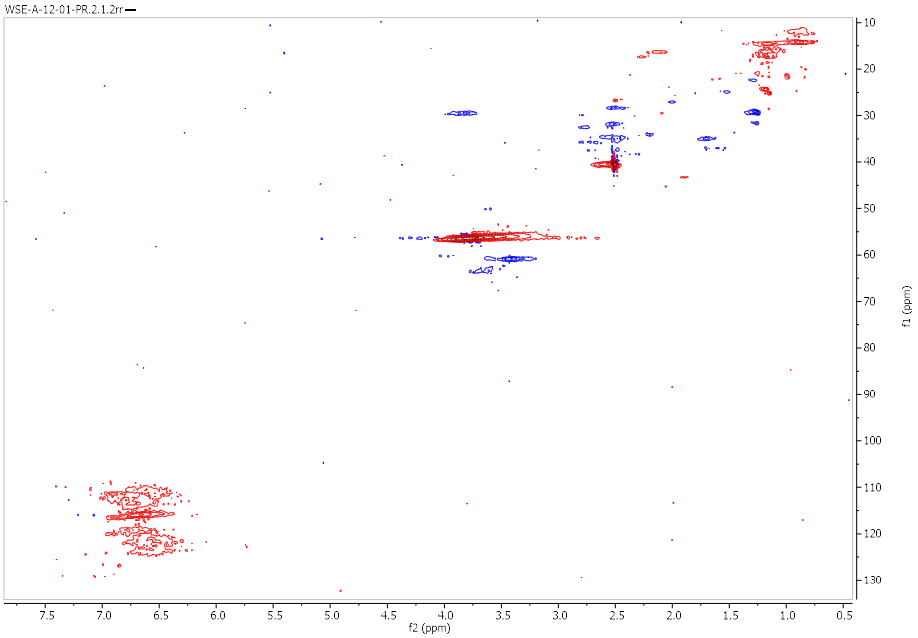

Figure S18. HSQC NMR spectra of KP phase obtained after reaction in water/EtOH mixture

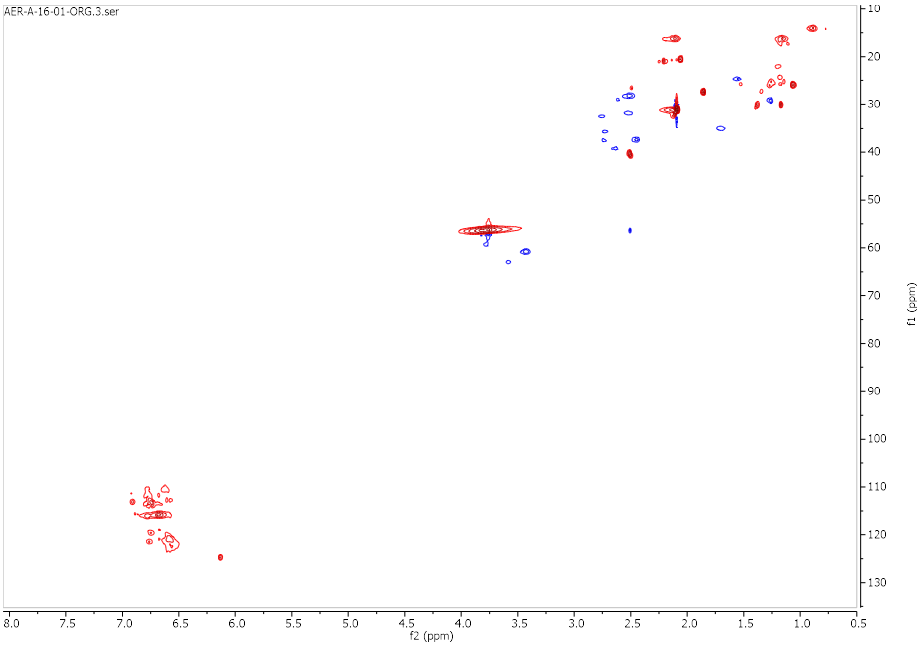

Figure S19. HSQC NMR spectra of OP phase obtained after reaction in water/iPrOH mixture

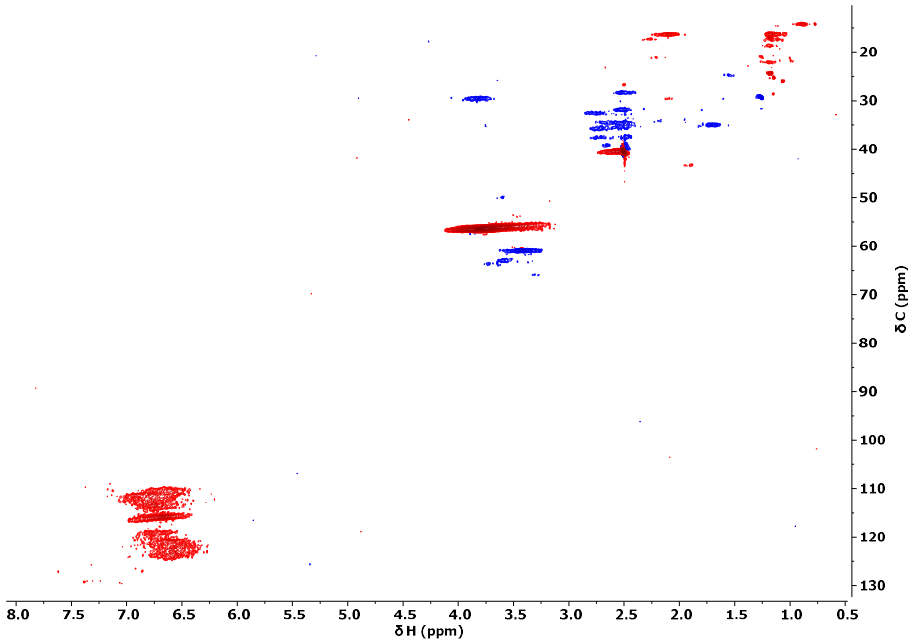

Figure S20. HSQC NMR spectra of KP phase obtained after reaction in water/iPrOH mixture

4. Monomeric products in 50/50 water/EtOH, 225°C

Compound	Pd/ZrO_2	$Pd(OAc)_2+ZrO_2$	$Pd(OAc)_2$	ZrO_2
ethylguaiacol	6.6	7.4	6.7	6.3
Guaiacol	6.4	6.7	6.8	6.1
Vanillyl methyl ketone	3.4	3.7	3.1	1.6
propylguaiacol	2	3.9	3.6	2.7
methylguaiacol	1.6	2.2	1.2	1.9
acetovanillone	1.6	1.4	1.9	1.5
iso-eugenol	1.2	1.4	1.4	0.8
Eugenol	0.5	0.7	0.7	0.7
homovanillyl alcohol	0.4	0.7	0.0	0.0
Vanillin	0.3	0.4	0.0	0.2
dimethoxyphenol	0.2	0.2	0.2	0.2
Phenol	0.2	0.2	0.3	0.6
Ethyl homovanillate	0.9	0.4	0.2	1.0
Ethyl hydroferulate	0.4	0.2	0.5	0.0
Total	25.7	29.37	26.6	23.38