

## Supporting Information

### **Facile Synthesis of $\text{CuCr}_2\text{O}_4$ Spinel Nanoparticles: A Recyclable Heterogeneous Catalyst for One Pot Hydroxylation of Benzene**

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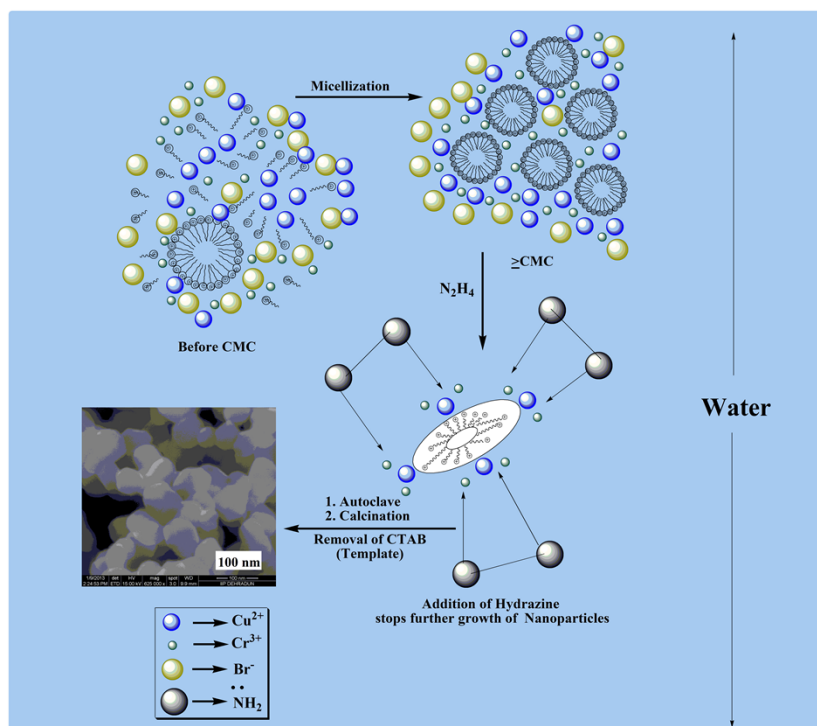
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Scheme S1. Formation of  $\text{CuCr}_2\text{O}_4$  Spinel Nanoparticles Catalyst.

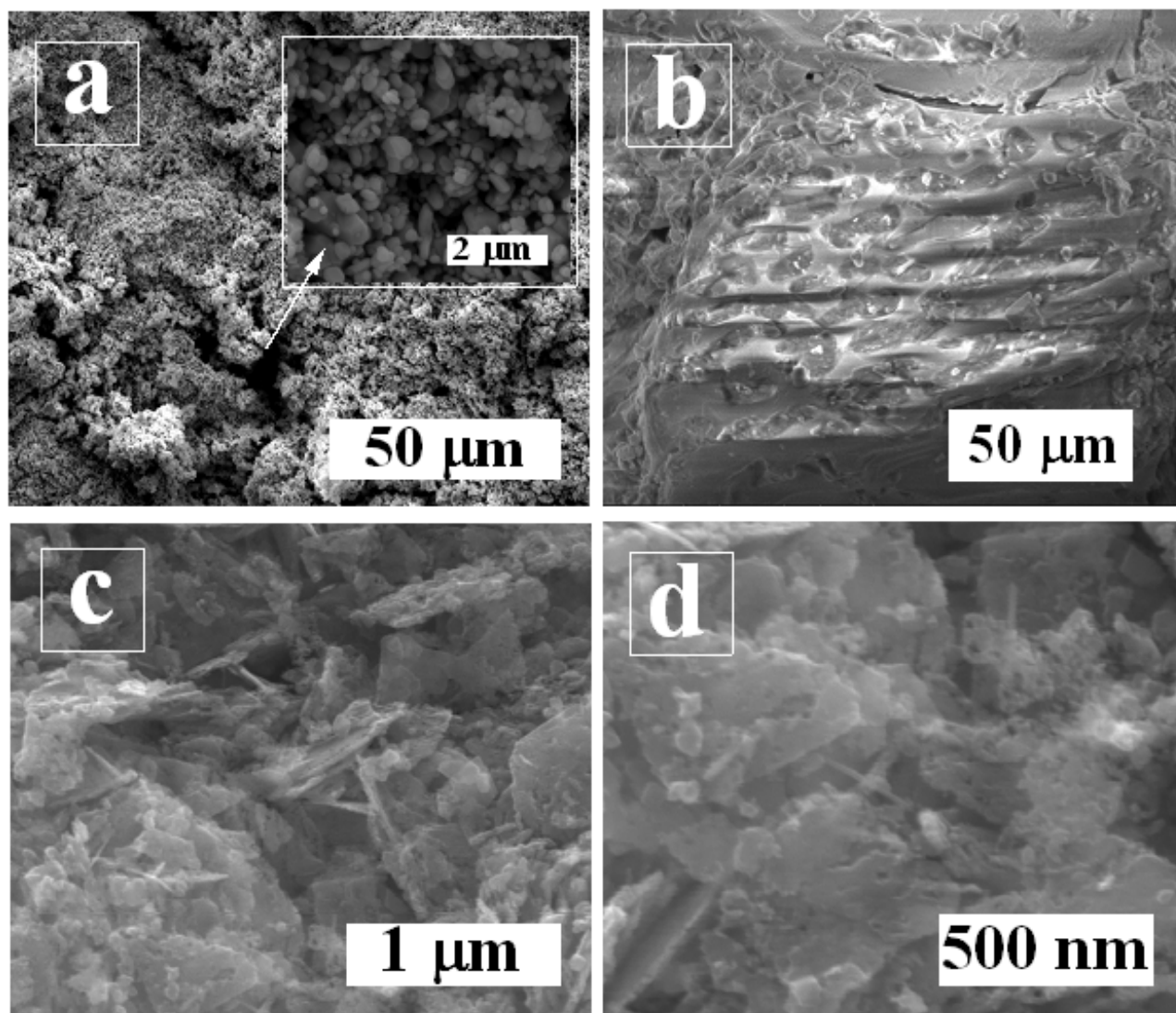


Figure S1. SEM images of Cu-Cr catalyst prepared by (a) impregnation method, (b) co-precipitation method and that of commercial catalyst at (c) low and (d) high resolution.

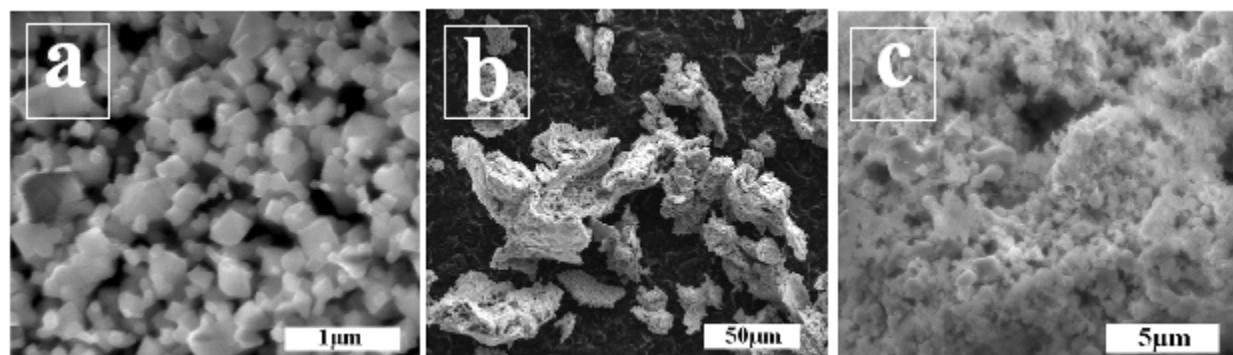


Figure S2. SEM image of the Cu-Cr samples without using (a)  $\text{NH}_4\text{OH}$  solution, (b) CTAB, (c) hydrazine.

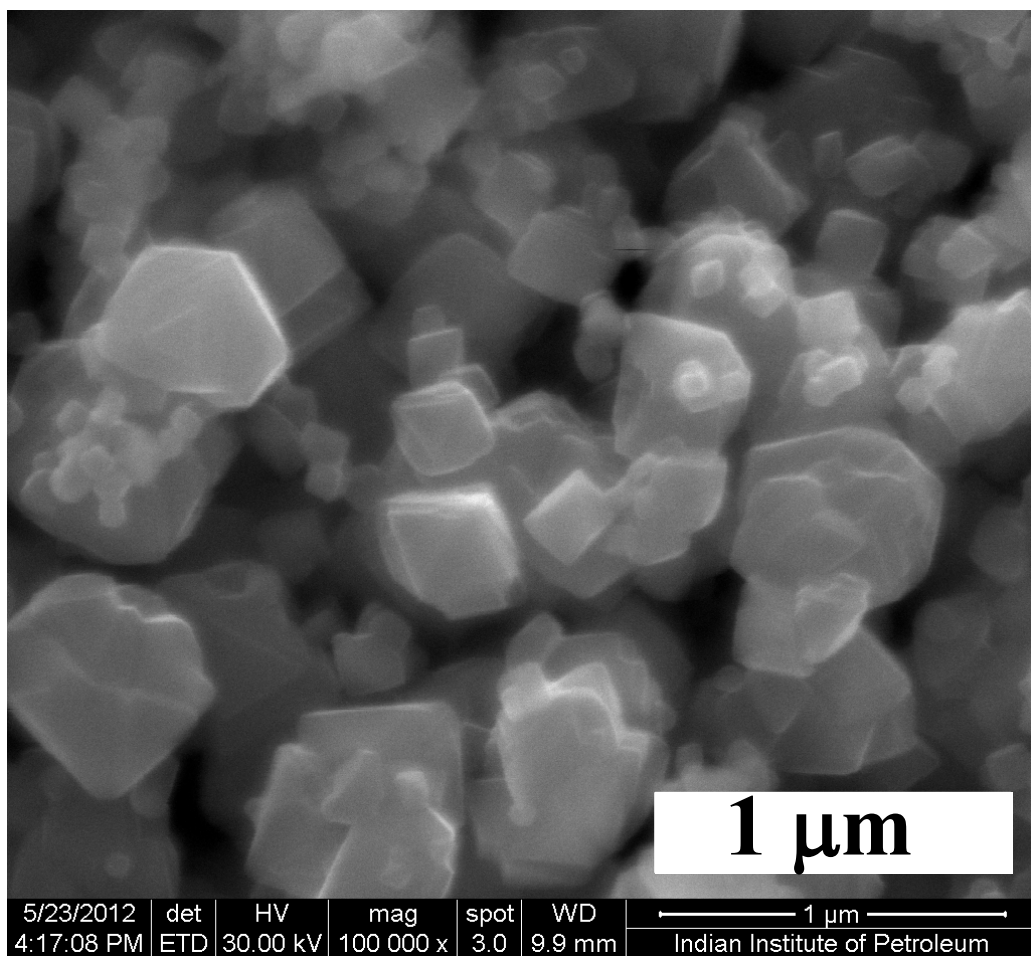


Figure S3. SEM image of the Cu-Cr samples prepared non-hydrothermally.

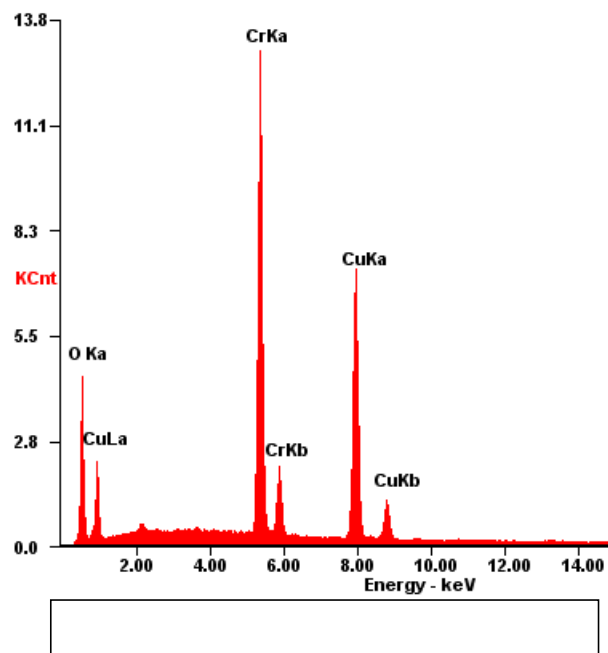


Figure S4. EDAX of the  $\text{CuCr}_2\text{O}_4$  spinel nanoparticles catalyst.

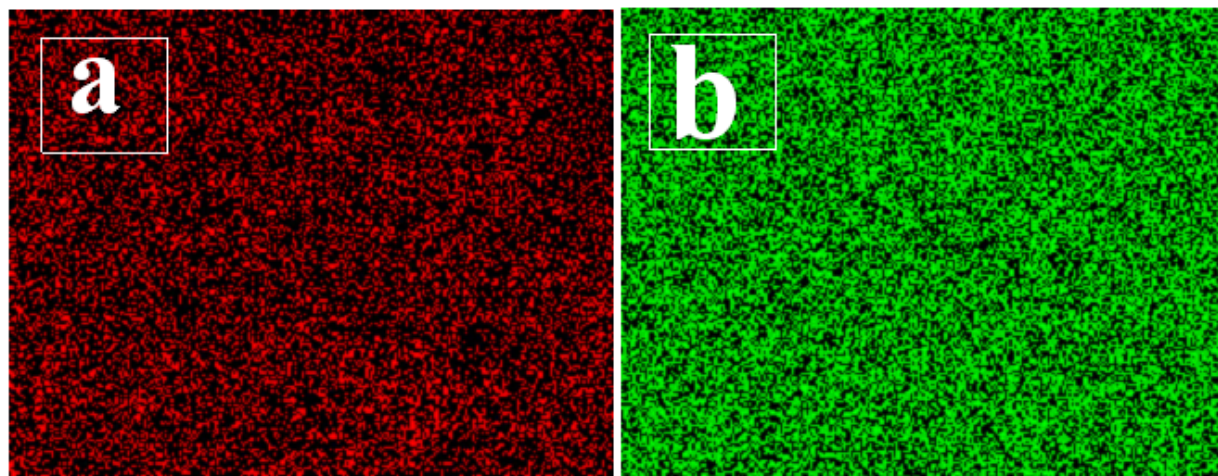


Figure S5. Elemental mapping of (a) Cu and (b) Cr atoms in the  $\text{CuCr}_2\text{O}_4$  spinel nanoparticles catalyst.

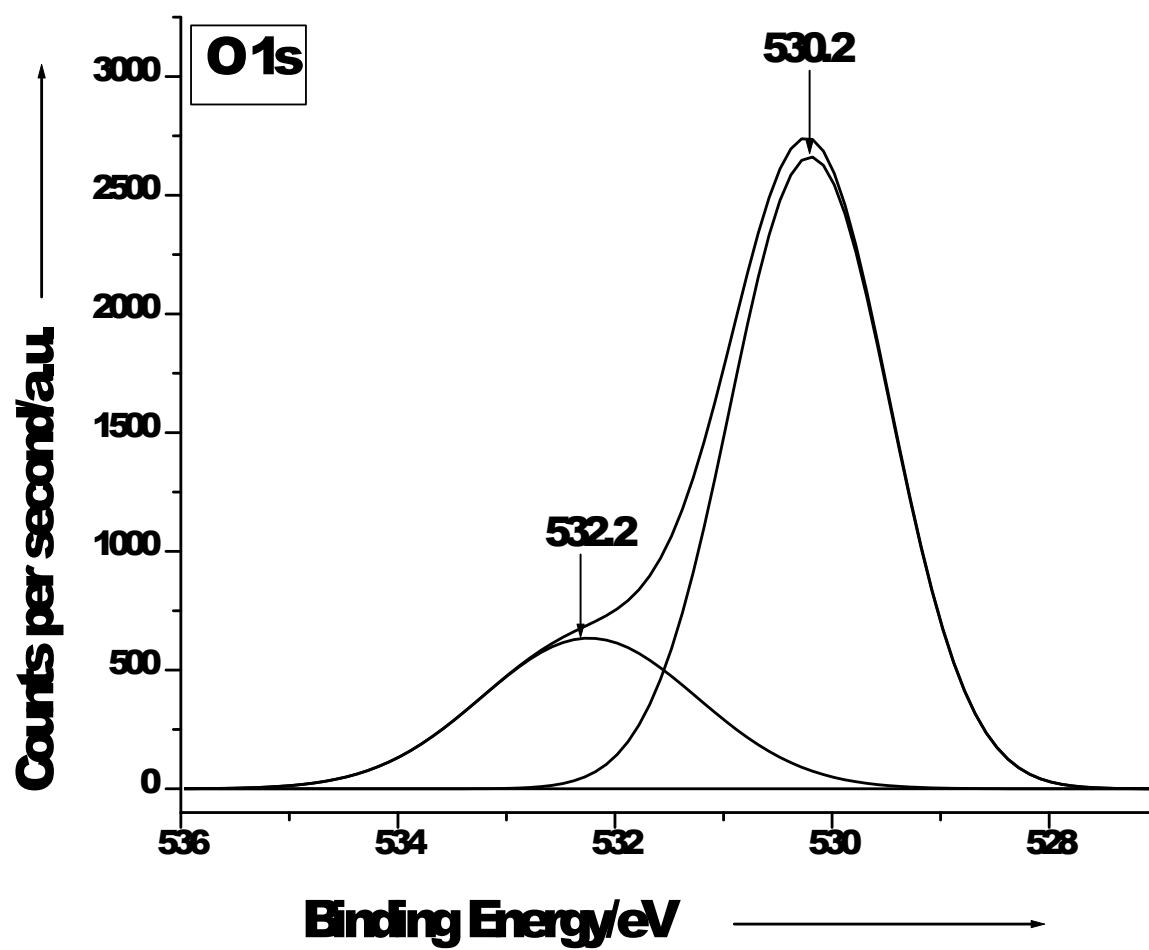


Figure S6. O1s core level spectra of CuCr<sub>2</sub>O<sub>4</sub> nano particles catalyst.

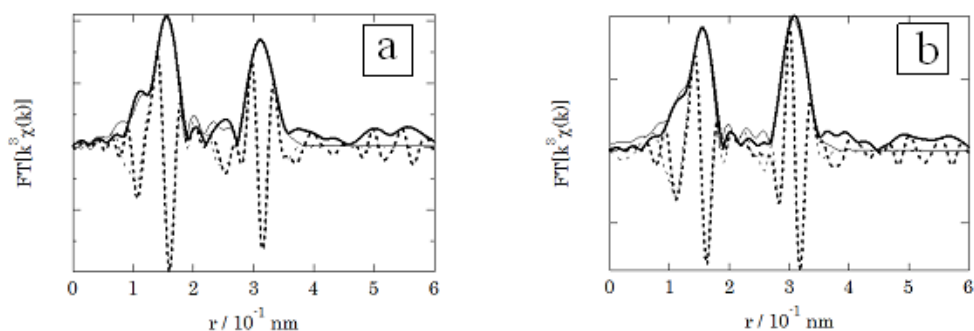


Figure S7. Fourier Transform of Cu-K edge EXAFS spectra of a) fresh catalyst and b) spent catalyst (after 5 recycles). Amplitude: solid curves; imaginary part: dotted curves; observed data: thick curves; fitting data: thin curves.

| Fresh Catalyst |          |      |       |            | Spent Catalyst (After 5 recycles) |          |      |       |            |
|----------------|----------|------|-------|------------|-----------------------------------|----------|------|-------|------------|
| No.            | Path     | CN   | R     | DW         | No.                               | Path     | CN   | R     | DW         |
| 1              | feff0001 | 4.00 | 1.886 | 1.683e-002 | 1                                 | feff0001 | 4.00 | 1.885 | 1.309e-002 |
| 2              | feff0002 | 4.00 | 3.140 | 2.198e-002 | 2                                 | feff0002 | 4.00 | 3.139 | 1.707e-002 |
| 3              | feff0003 | 4.00 | 3.245 | 9.406e-003 | 3                                 | feff0003 | 4.00 | 3.243 | 7.311e-003 |
| 4              | feff0004 | 8.00 | 3.362 | 2.948e-002 | 4                                 | feff0004 | 8.00 | 3.368 | 2.293e-002 |
| 5              | feff0005 | 8.00 | 3.458 | 9.499e-003 | 5                                 | feff0005 | 8.00 | 3.464 | 7.379e-003 |
| 6              | feff0006 | 4.00 | 3.523 | 3.152e-002 | 6                                 | feff0006 | 4.00 | 3.529 | 2.450e-002 |
| 7              | feff0007 | 4.00 | 3.536 | 8.558e-003 | 7                                 | feff0007 | 4.00 | 3.542 | 6.647e-003 |
| 8              | feff0008 | 8.00 | 3.538 | 2.230e-002 | 8                                 | feff0008 | 8.00 | 3.544 | 1.731e-002 |
| 9              | feff0009 | 8.00 | 3.541 | 1.231e-002 | 9                                 | feff0009 | 8.00 | 3.547 | 9.572e-003 |
| 10             | CuO      | 2.02 | 1.938 | 2.233e-003 | 10                                | CuO      | 2.33 | 1.939 | 2.933e-003 |

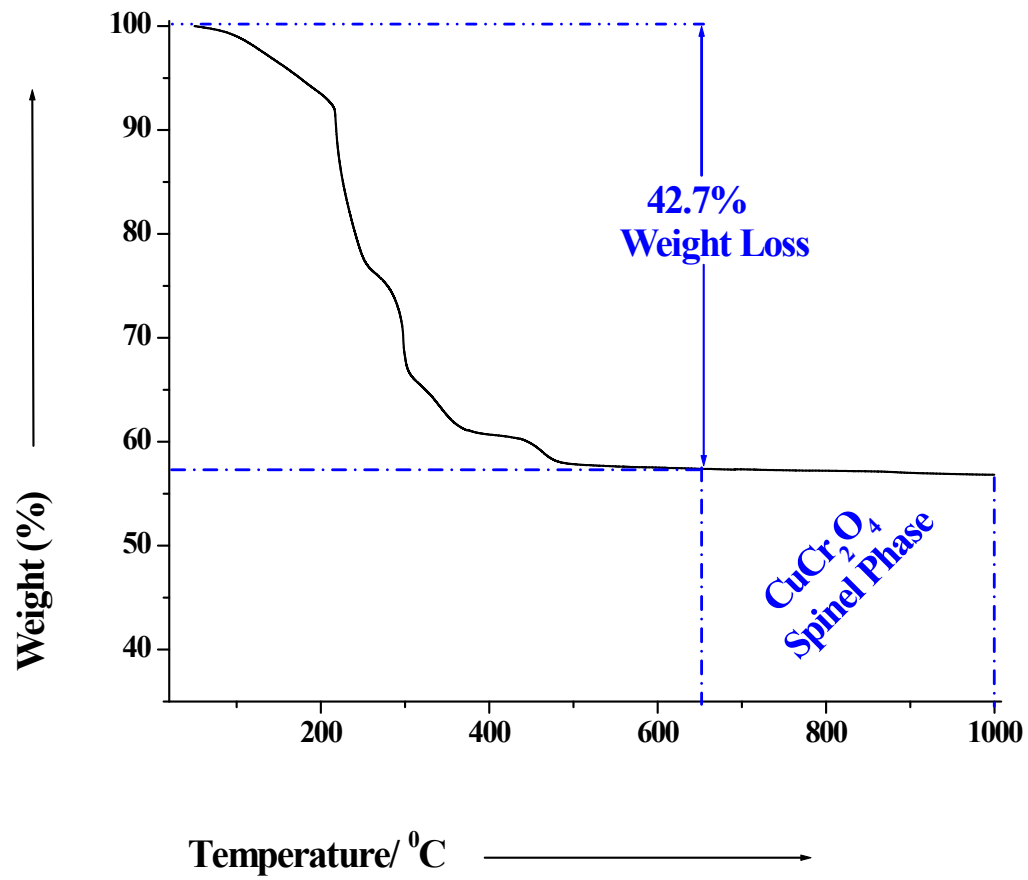


Figure S8. TGA analysis of uncalcined  $\text{CuCr}_2\text{O}_4$  spinel nanoparticles catalyst.

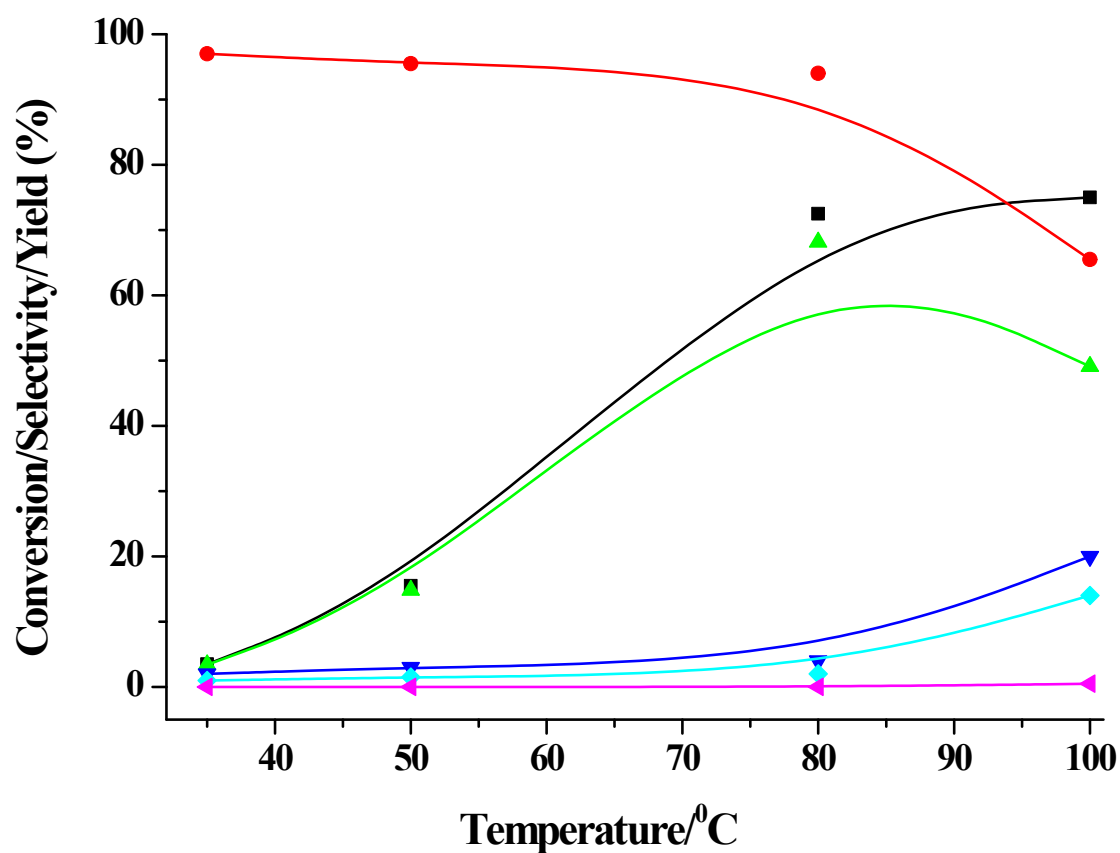


Figure S9. Effect of temperature on benzene hydroxylation.

[■] Conversion of benzene; [●] Selectivity to phenol; [▲] Yield of phenol; [▼] Selectivity to catechol [◆] Selectivity to hydroquinone; [◄] Selectivity to biphenyl.

Reaction Condition: benzene = 1g; catalyst = 0.1g; benzene: H<sub>2</sub>O<sub>2</sub> mole ratio = 1:5; time = 12 h

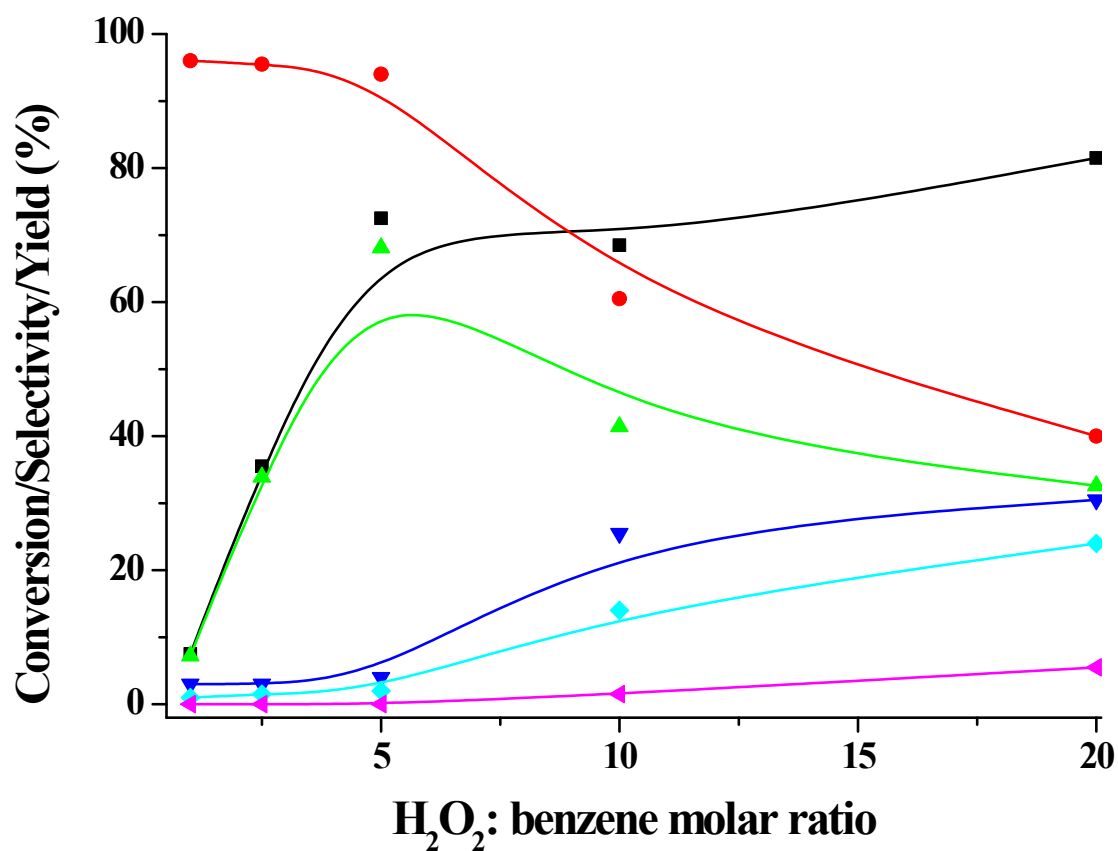


Figure S10. Effect of H<sub>2</sub>O<sub>2</sub>: benzene molar ratio on benzene hydroxylation.

[■] Conversion of benzene; [●] Selectivity to phenol; [▲] Yield of phenol; [▼] Selectivity to catechol [◆] Selectivity to hydroquinone; [◀] Selectivity to biphenyl.

Reaction Condition: benzene = 1g; catalyst = 0.1g; temperature = 80 °C; time = 12 h.

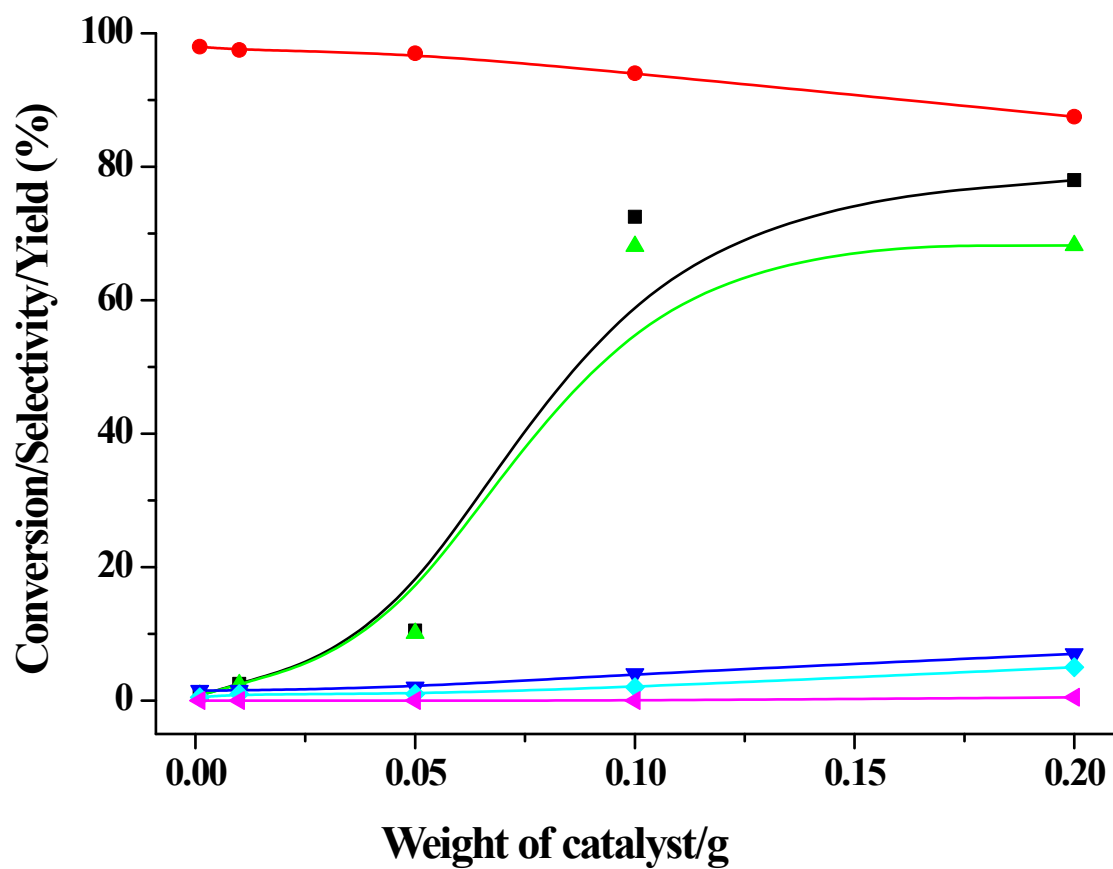


Figure S11. Effect of weight of catalyst on benzene hydroxylation.

[■] Conversion of benzene; [●] Selectivity to phenol; [▲] Yield of phenol; [▼] Selectivity to catechol [◆] Selectivity to hydroquinone; [◀] Selectivity to biphenyl.

Reaction Condition: benzene =1g; benzene: H<sub>2</sub>O<sub>2</sub> mole ratio =1:5; temperature = 80 °C; time= 12 h.

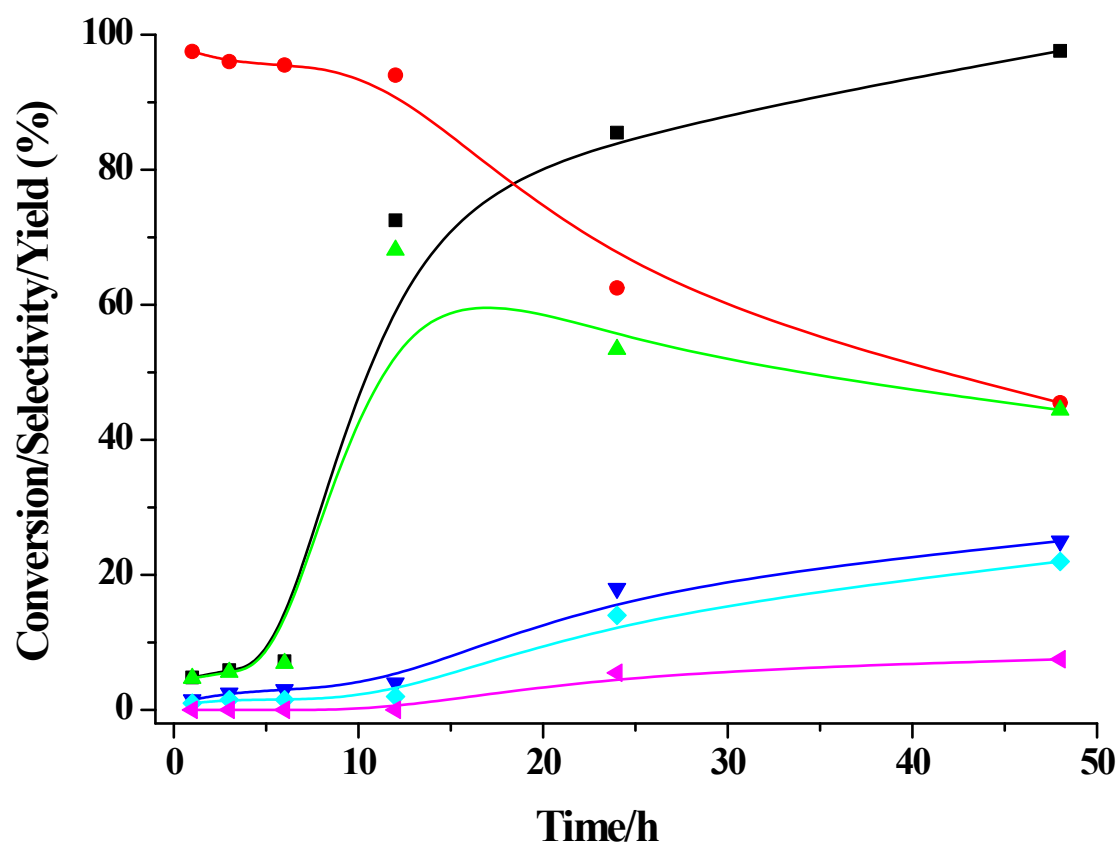


Figure S12. Effect of time on benzene hydroxylation.

[■] Conversion of benzene; [●] Selectivity to phenol; [▲] Yield of phenol; [▼] Selectivity to catechol [◆] Selectivity to hydroquinone; [◄] Selectivity to biphenyl.

Reaction Condition: benzene = 1g; benzene:  $\text{H}_2\text{O}_2$  mole ratio = 1:5; temperature = 80 °C.