SUPPORTING INFORMATION

One-Pot Tandem Catalysis: Green Synthesis of β-Pinene Derivatives with MgO and Mesoporous Catalysts

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1. Synthesis of materials

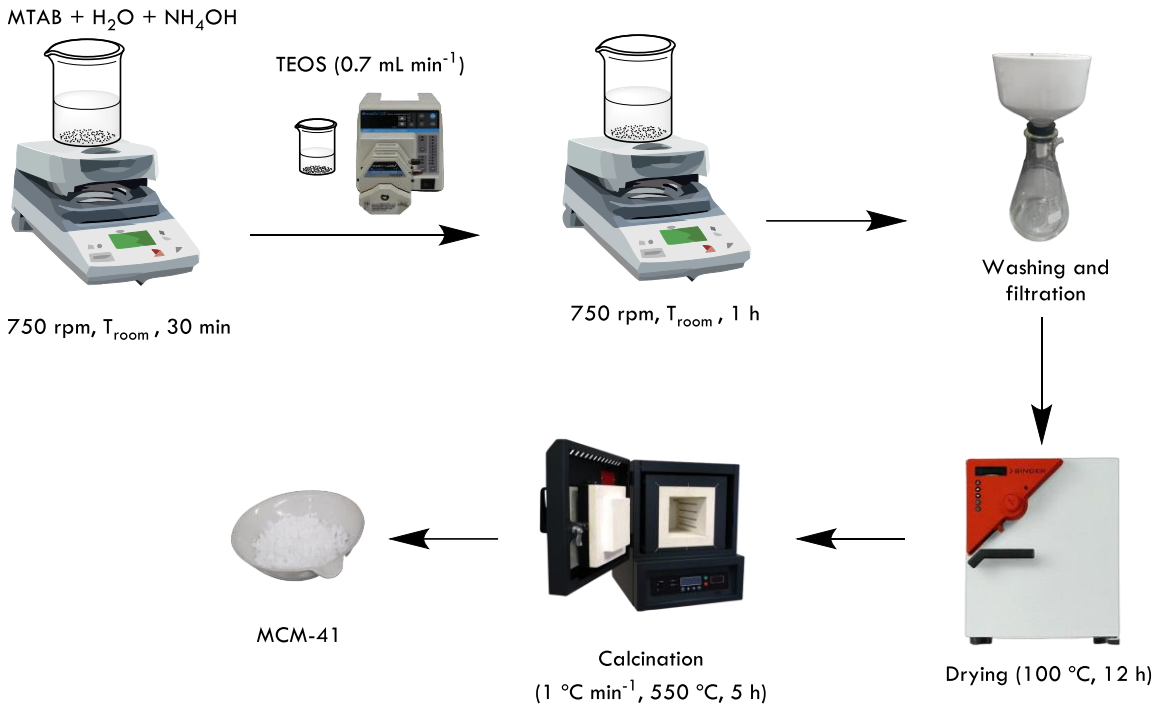


Figure S1. Scheme of the synthesis of MCM-41 support.

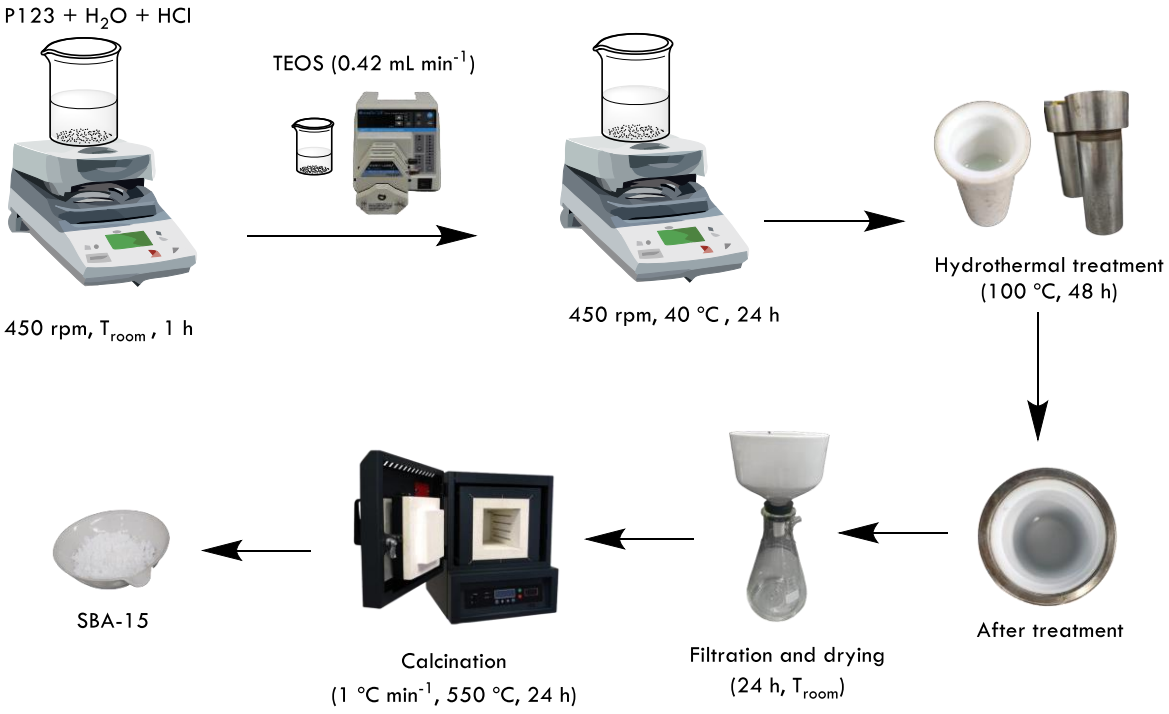


Figure S2. Scheme of the synthesis of SBA-15 support.

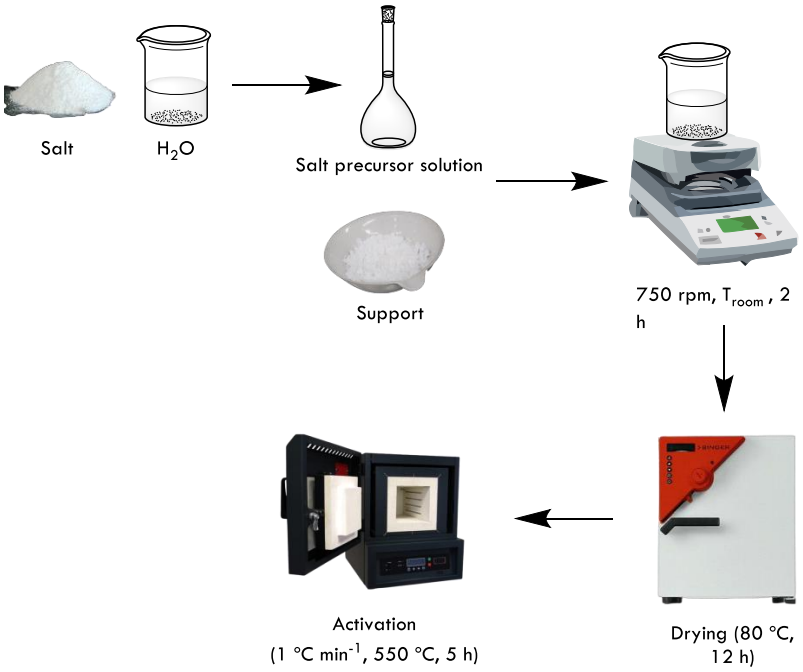


Figure S3. Scheme of the wetness impregnation procedure.

2. TEM images

2.1. MCM-41

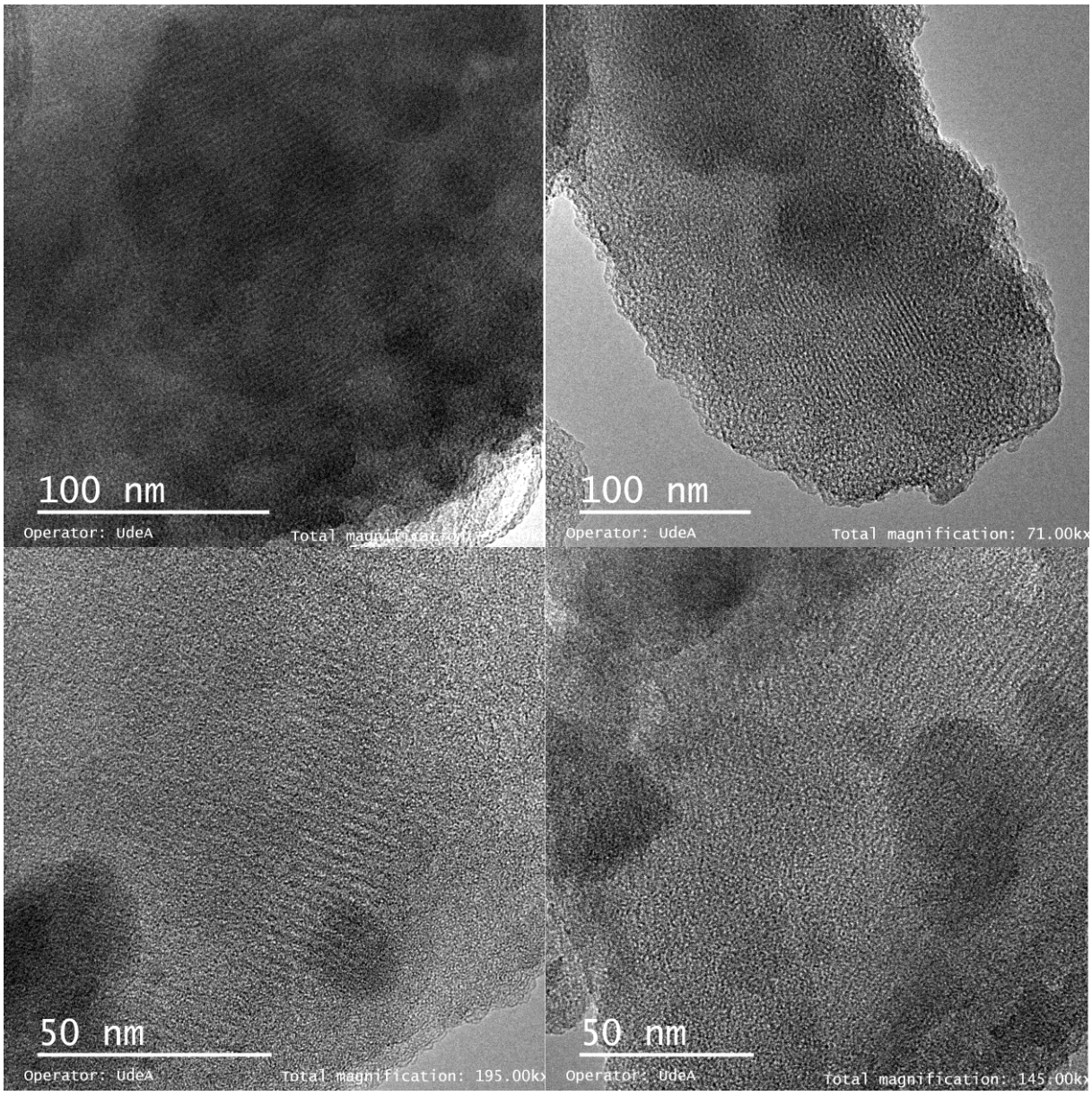


Figure S4. TEM images of MCM-41 support.

2.2. SnM1

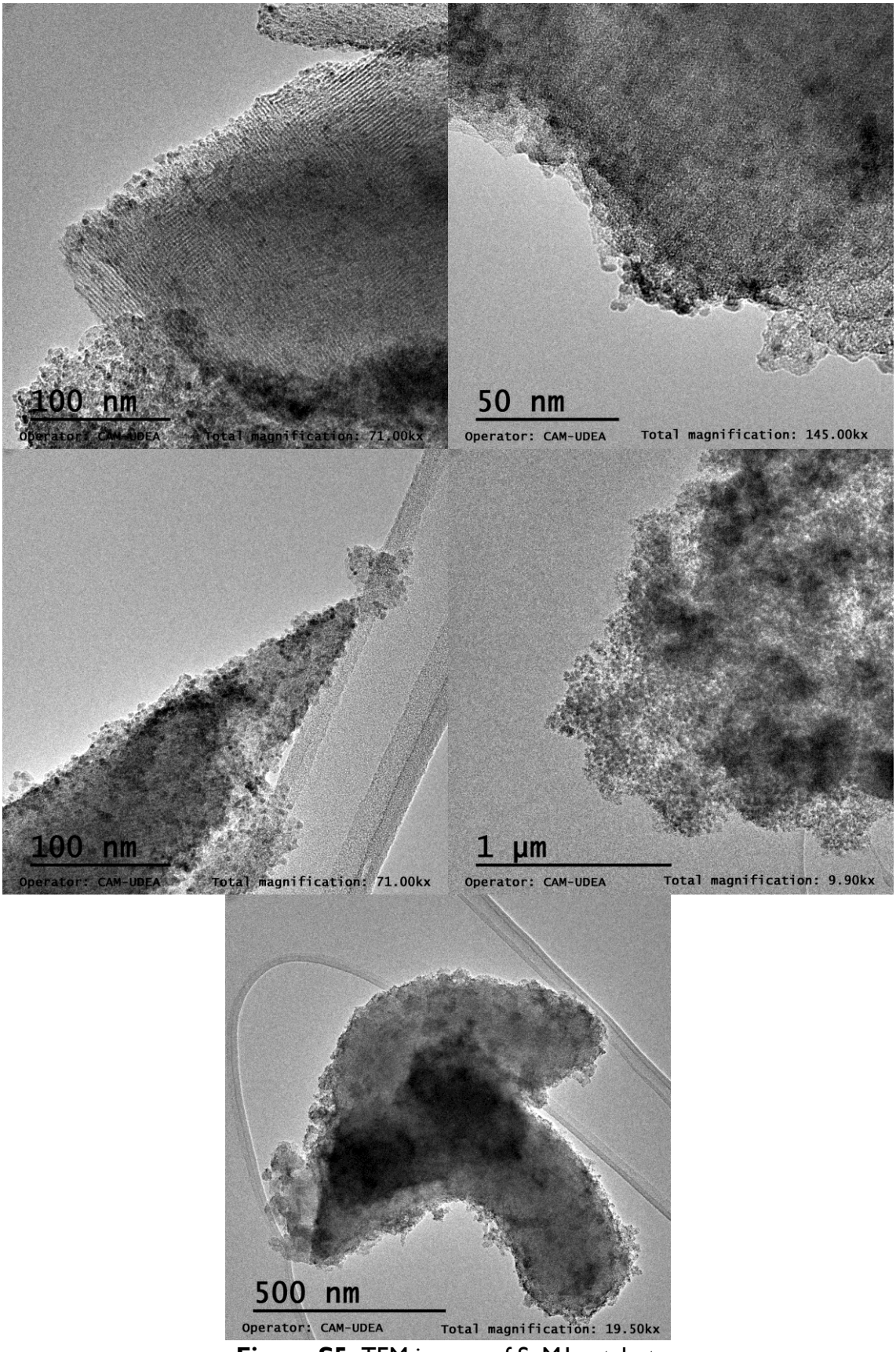


Figure S5. TEM images of SnM1 catalyst.

2.3. SnM2

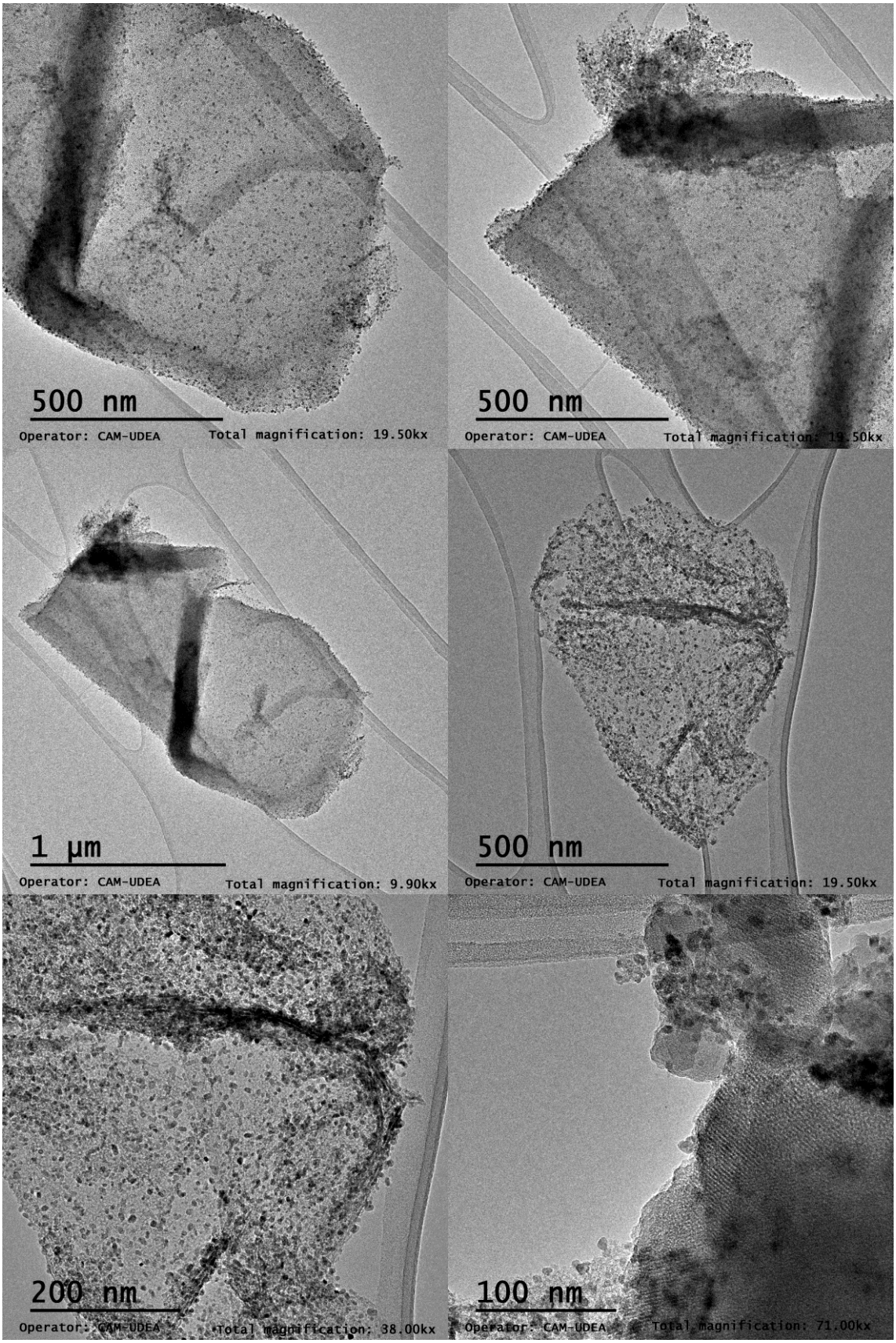


Figure S6. TEM images of SnM2 catalyst.

2.4. FeM1

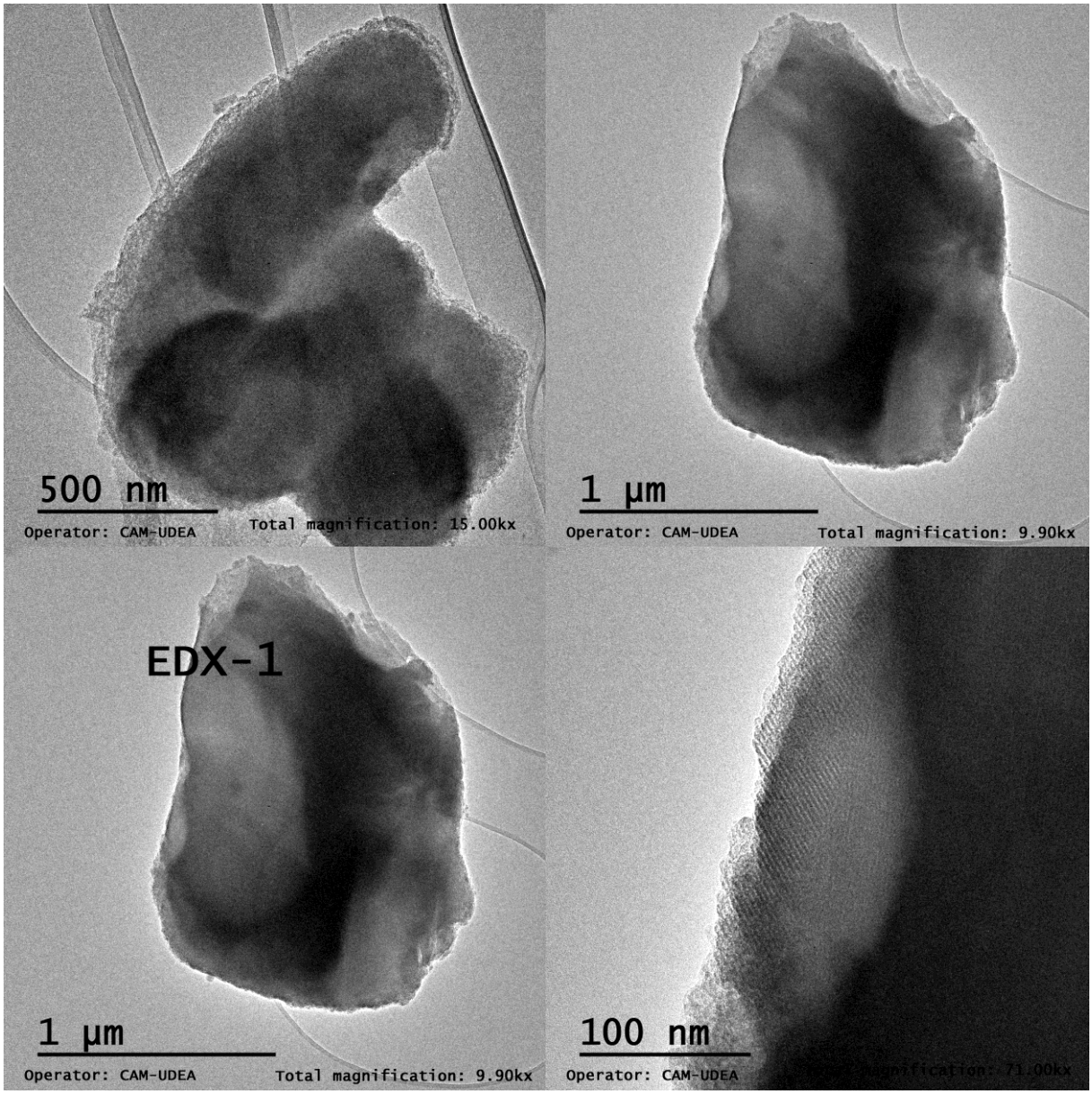


Figure S7. TEM images of FeM1 catalyst.

2.5. CuM1

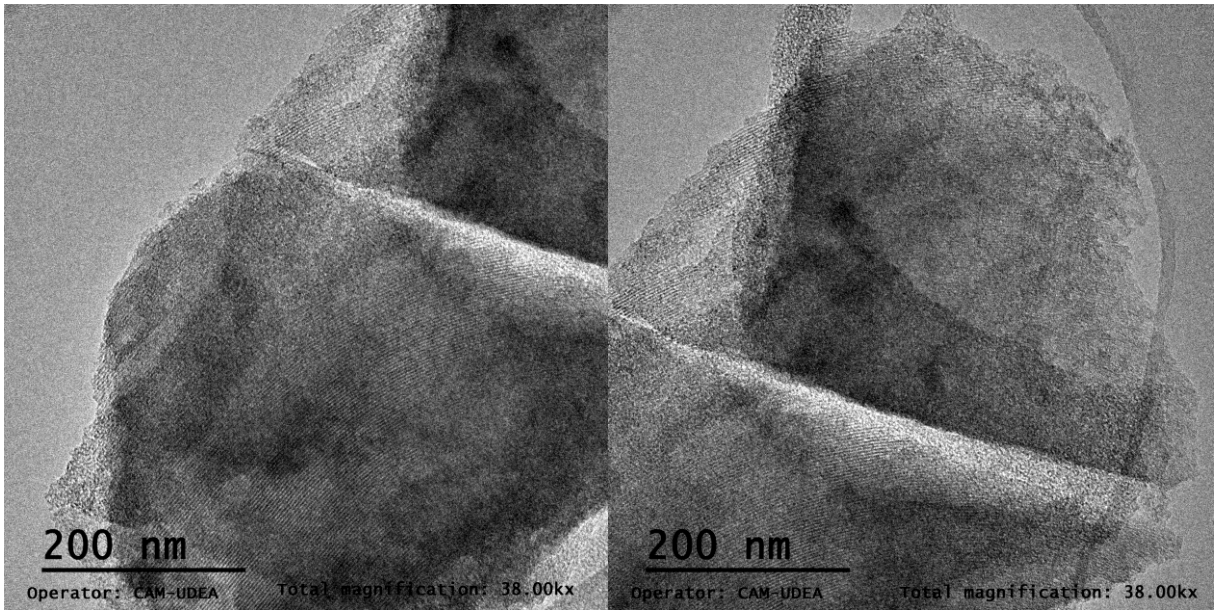


Figure S8. TEM images of CuM1 catalyst.

2.6. CoM1

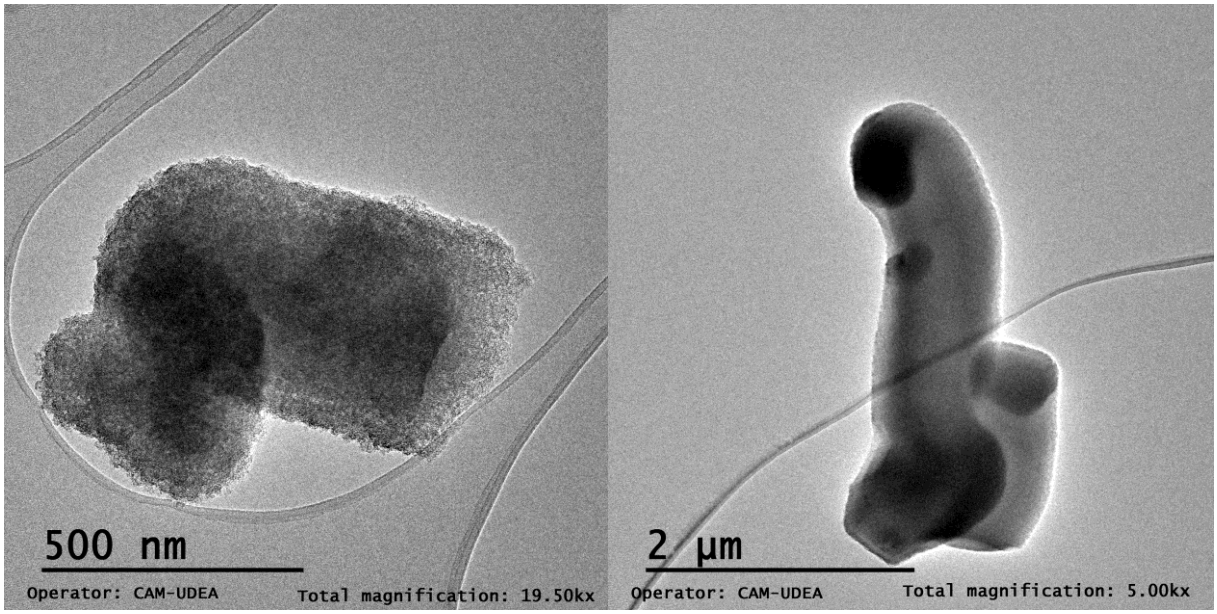


Figure S9. TEM images of CoM1 catalyst.

2.7. SnS1

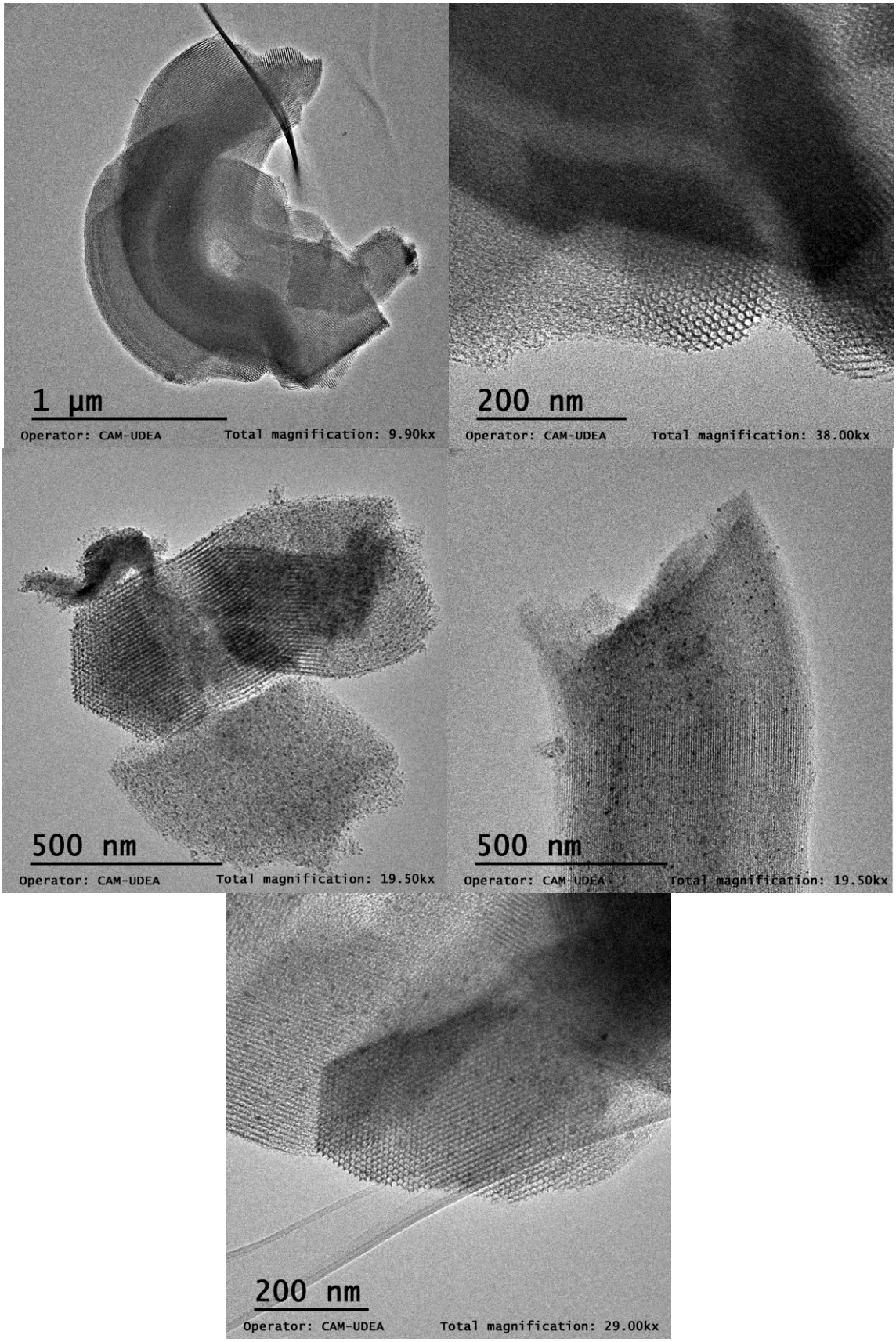


Figure S10. TEM images of SnS1 catalyst.

2.8. SnS2

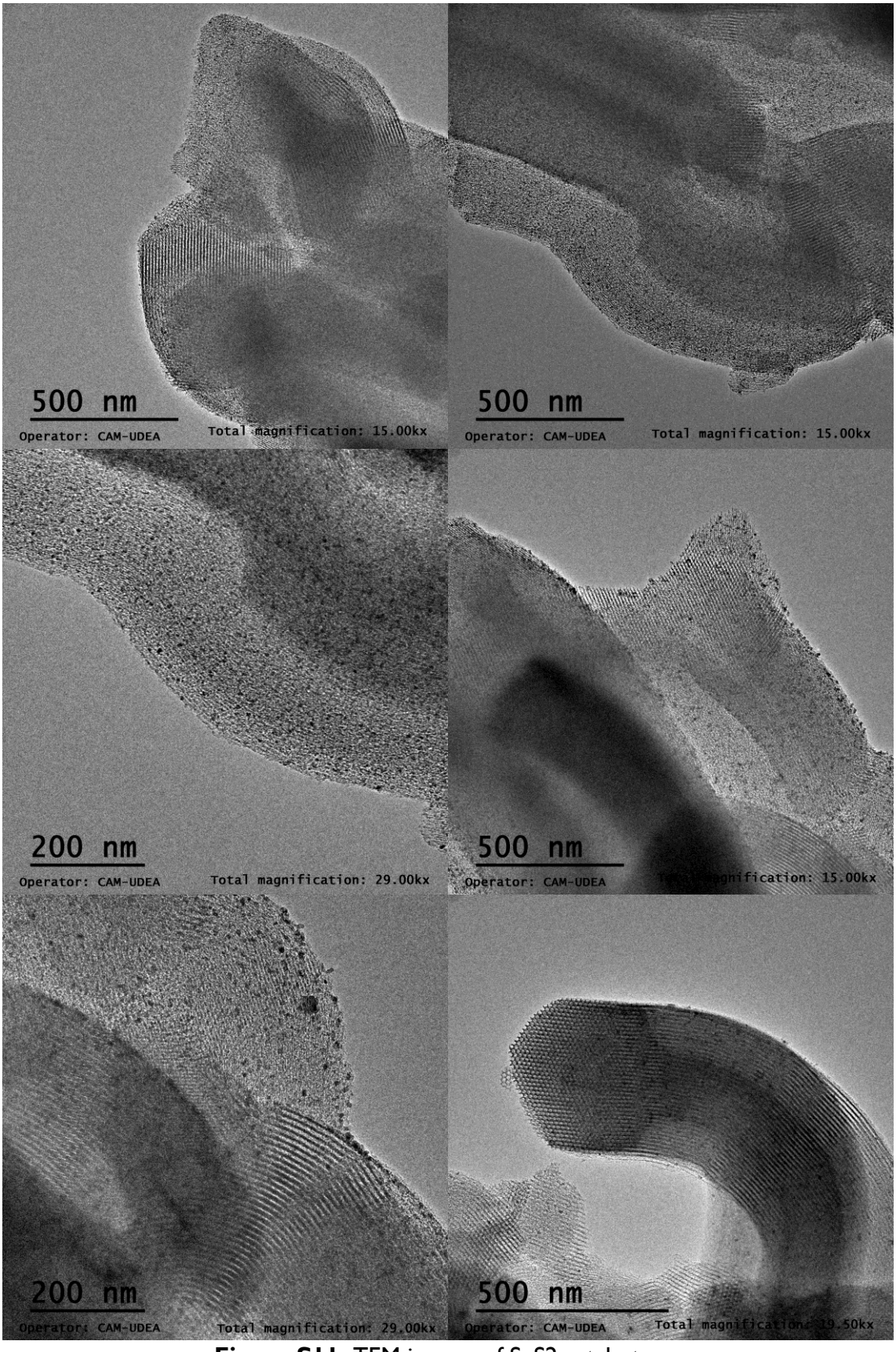


Figure S11. TEM images of SnS2 catalyst.

2.9. FeS1

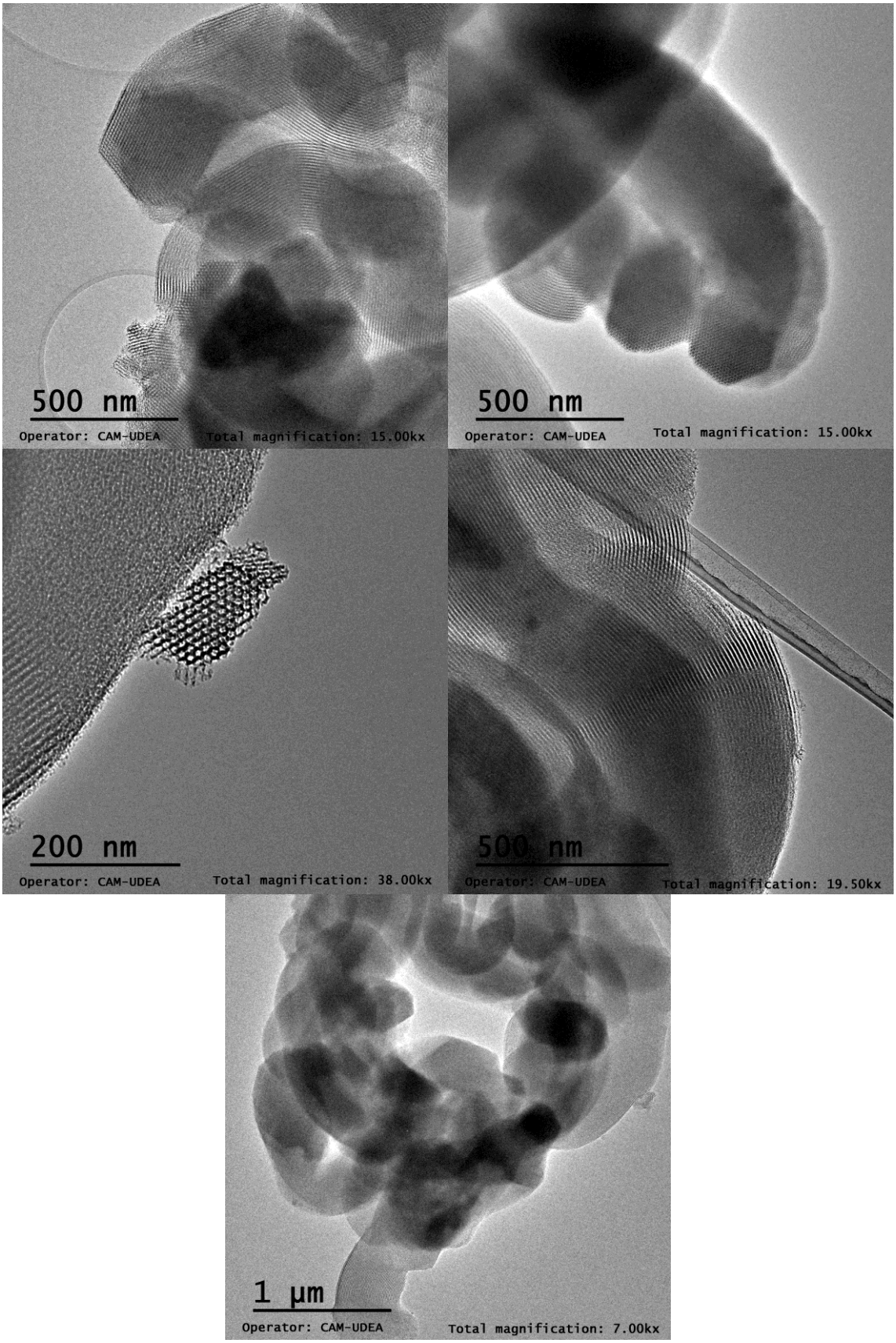


Figure S12. TEM images of FeS1 catalyst.

2.10. FeS2

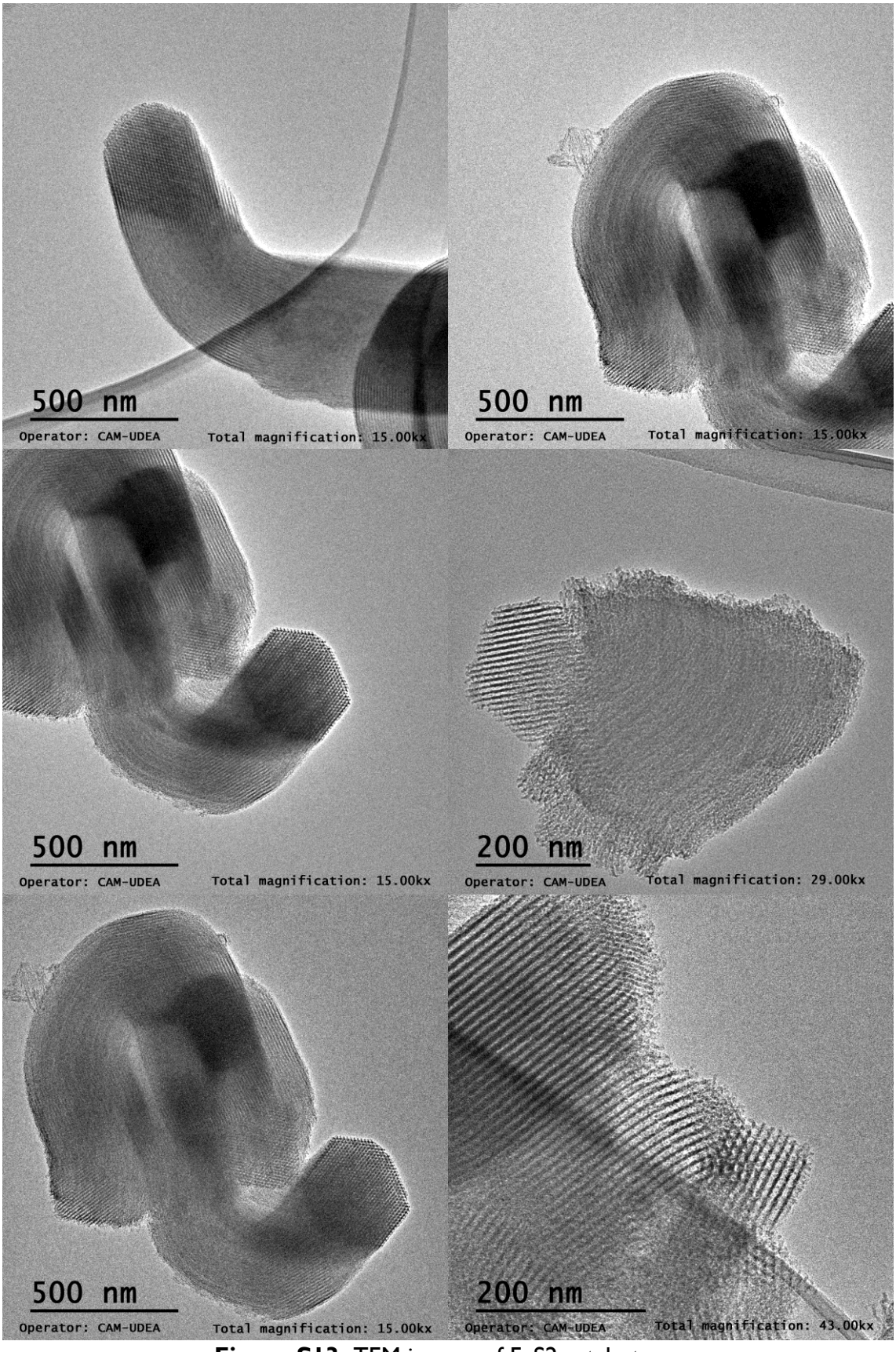


Figure S13. TEM images of FeS2 catalyst.

2.11. CuS1

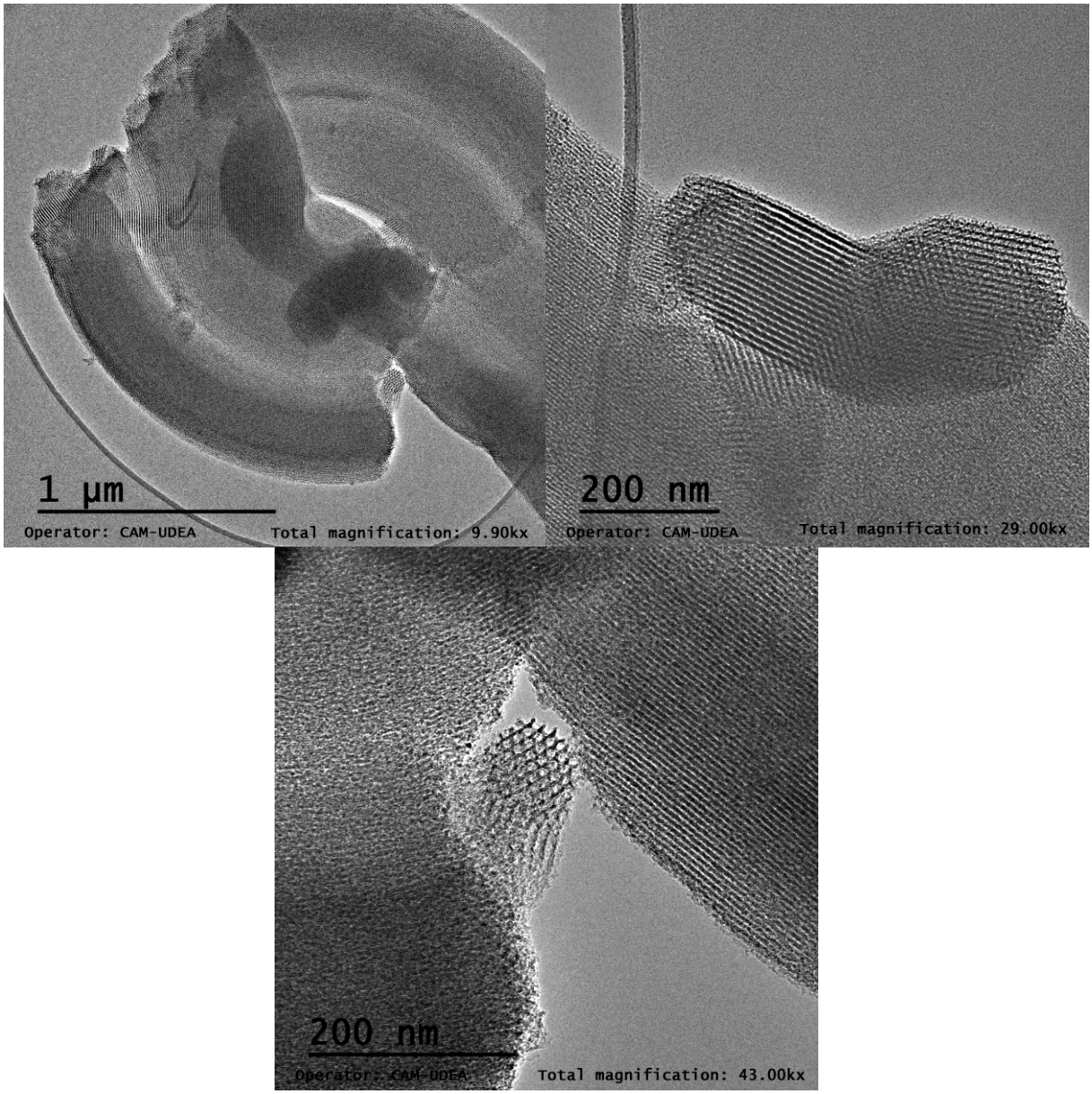


Figure S14. TEM images of CuS1 catalyst.

2.12. CoS1

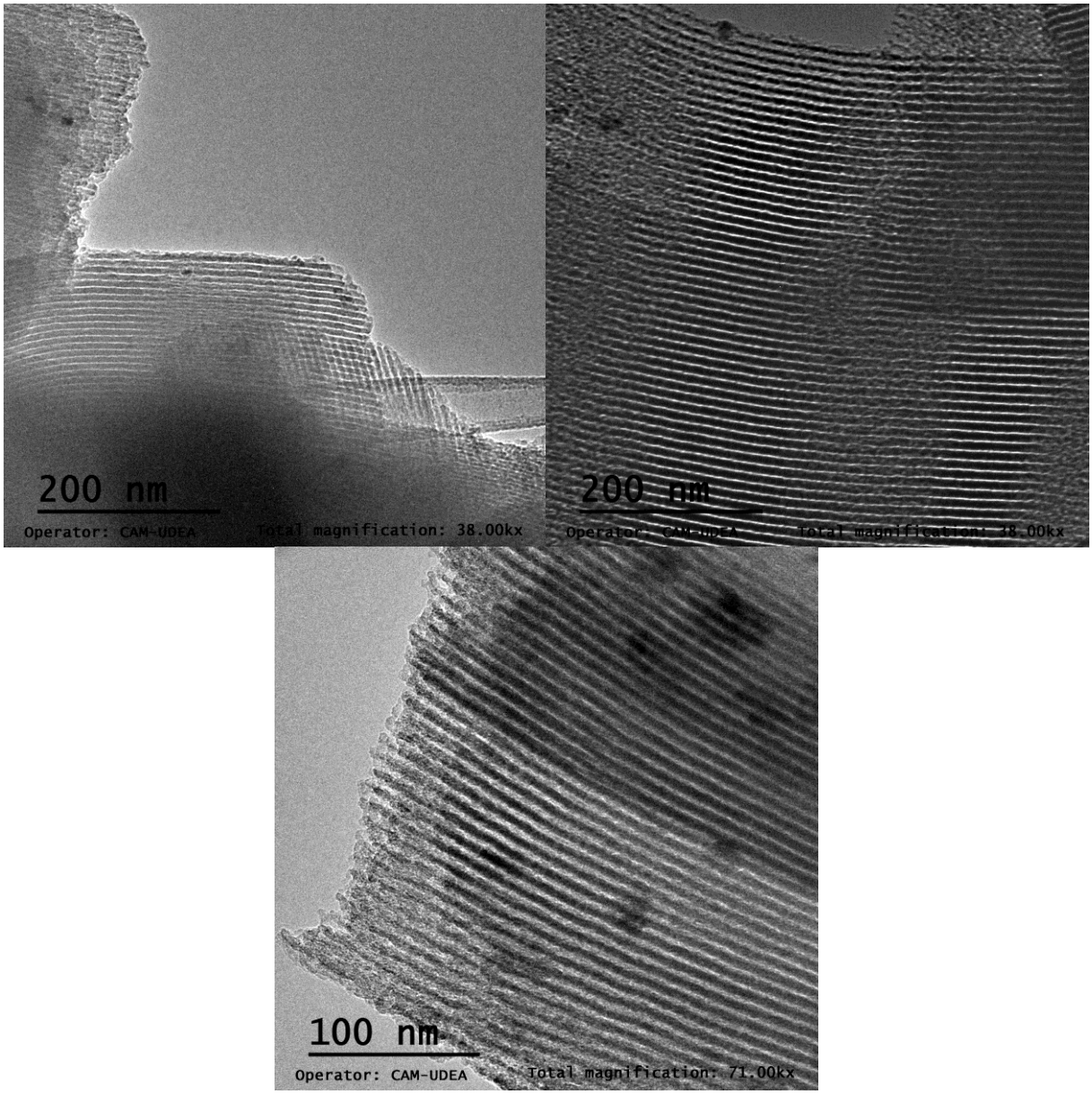


Figure S15. TEM images of CoS1 catalyst.

3. Elemental maps

3.1. FeM1

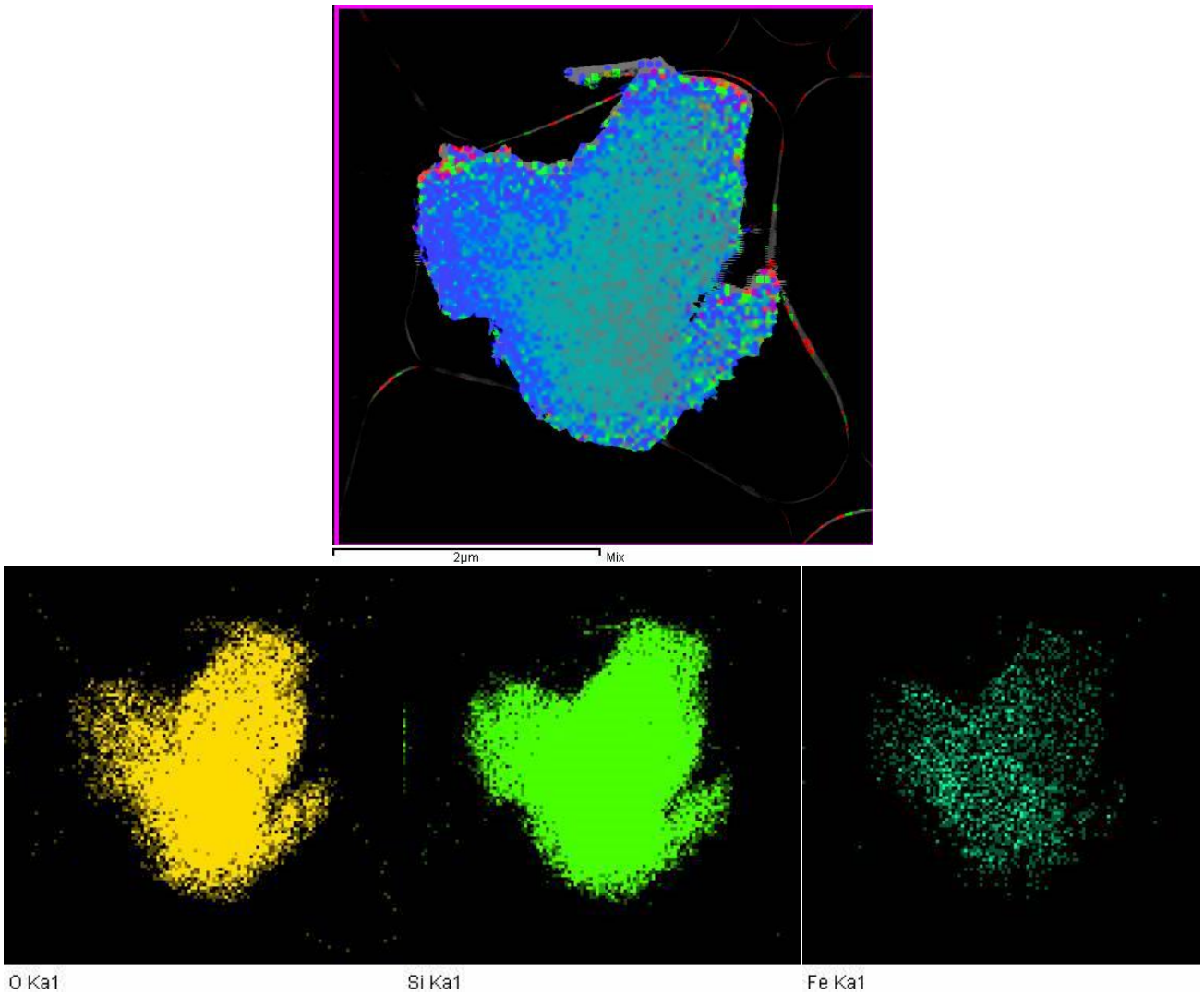


Figure S16. Elemental maps of the FeM1 catalyst.

3.2. SnS1

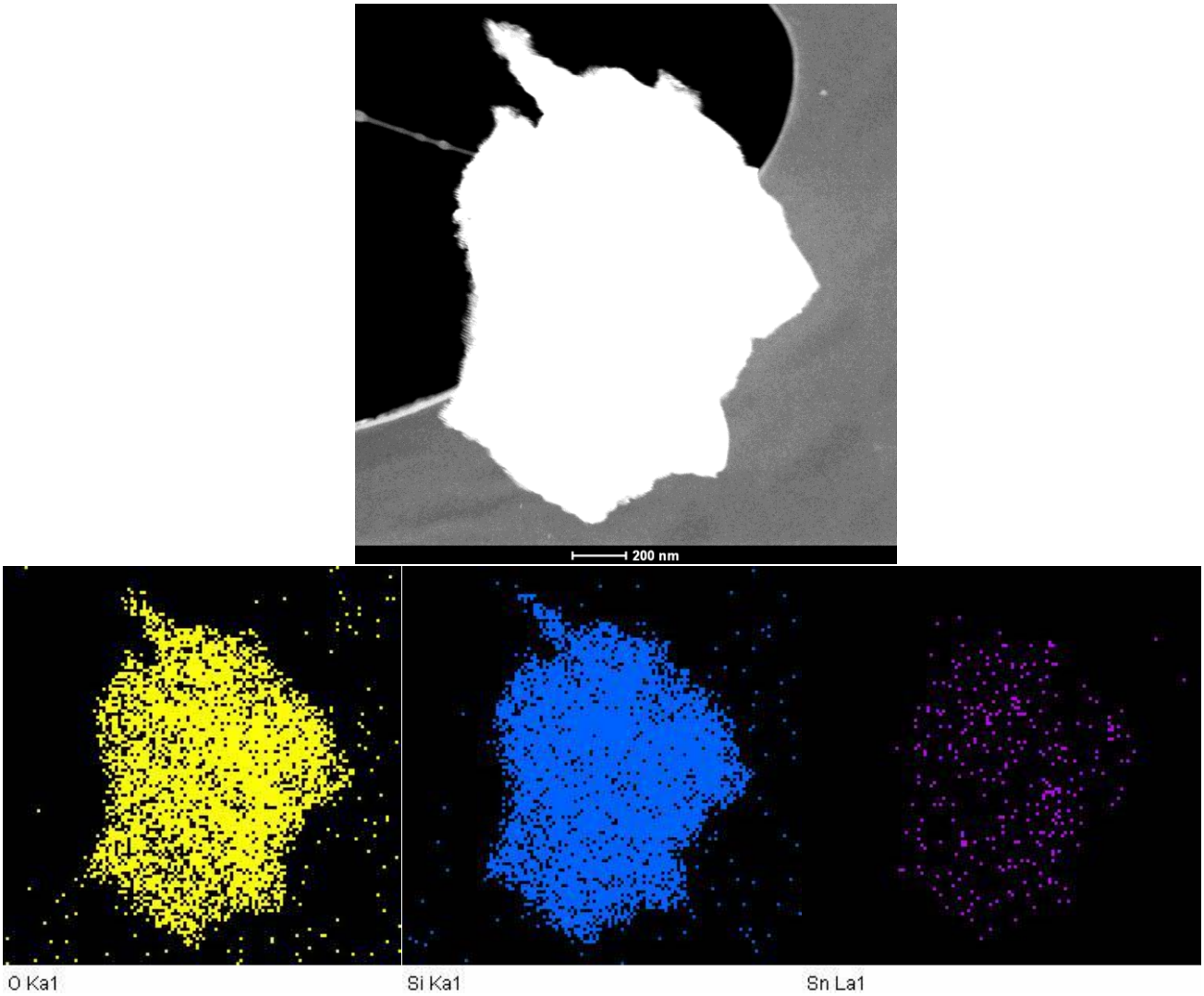


Figure S17. Elemental maps of the SnS1 catalyst.

3.3. CuS1

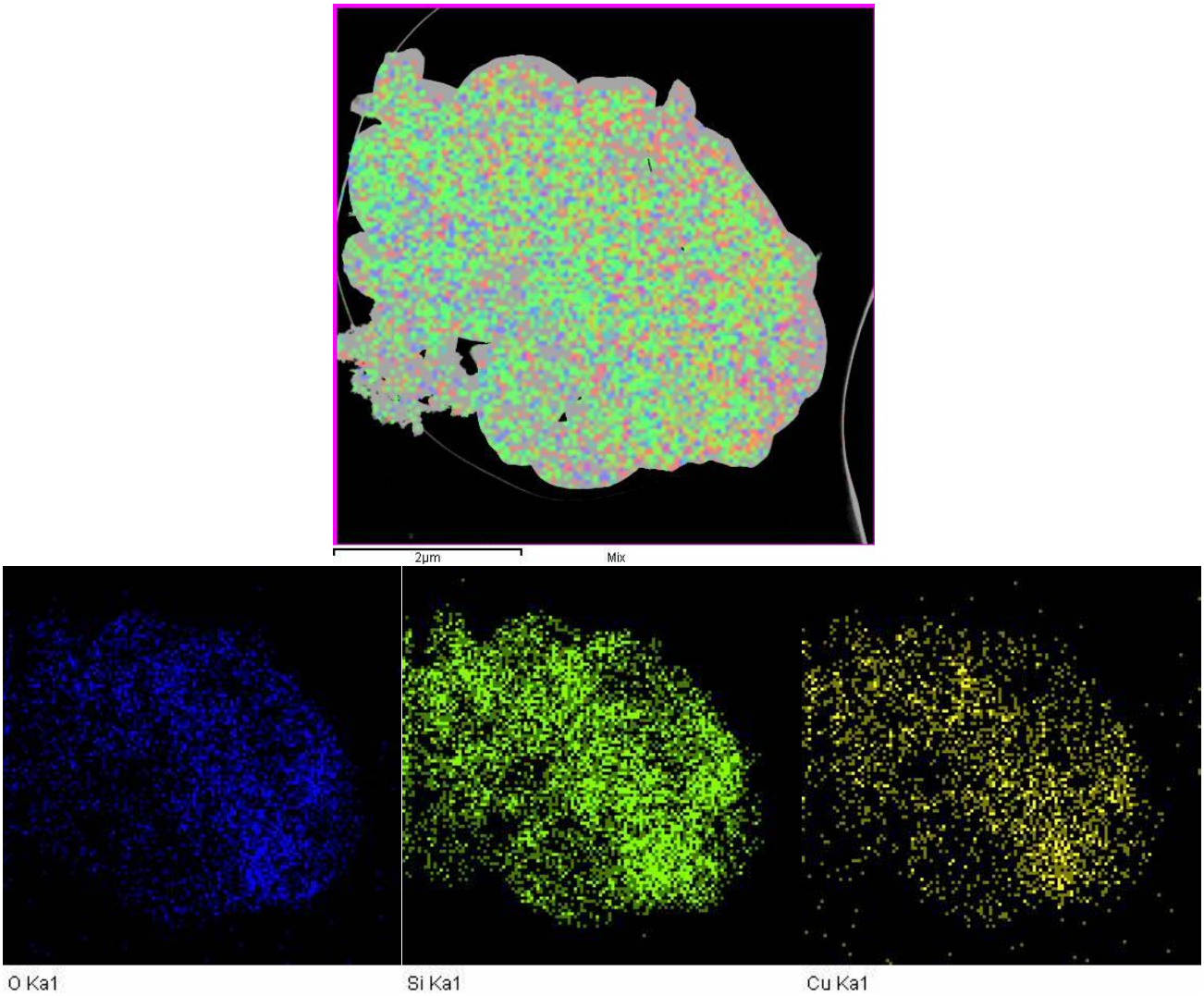


Figure S18. Elemental maps of the CuS1 catalyst.

3.4. CoS1

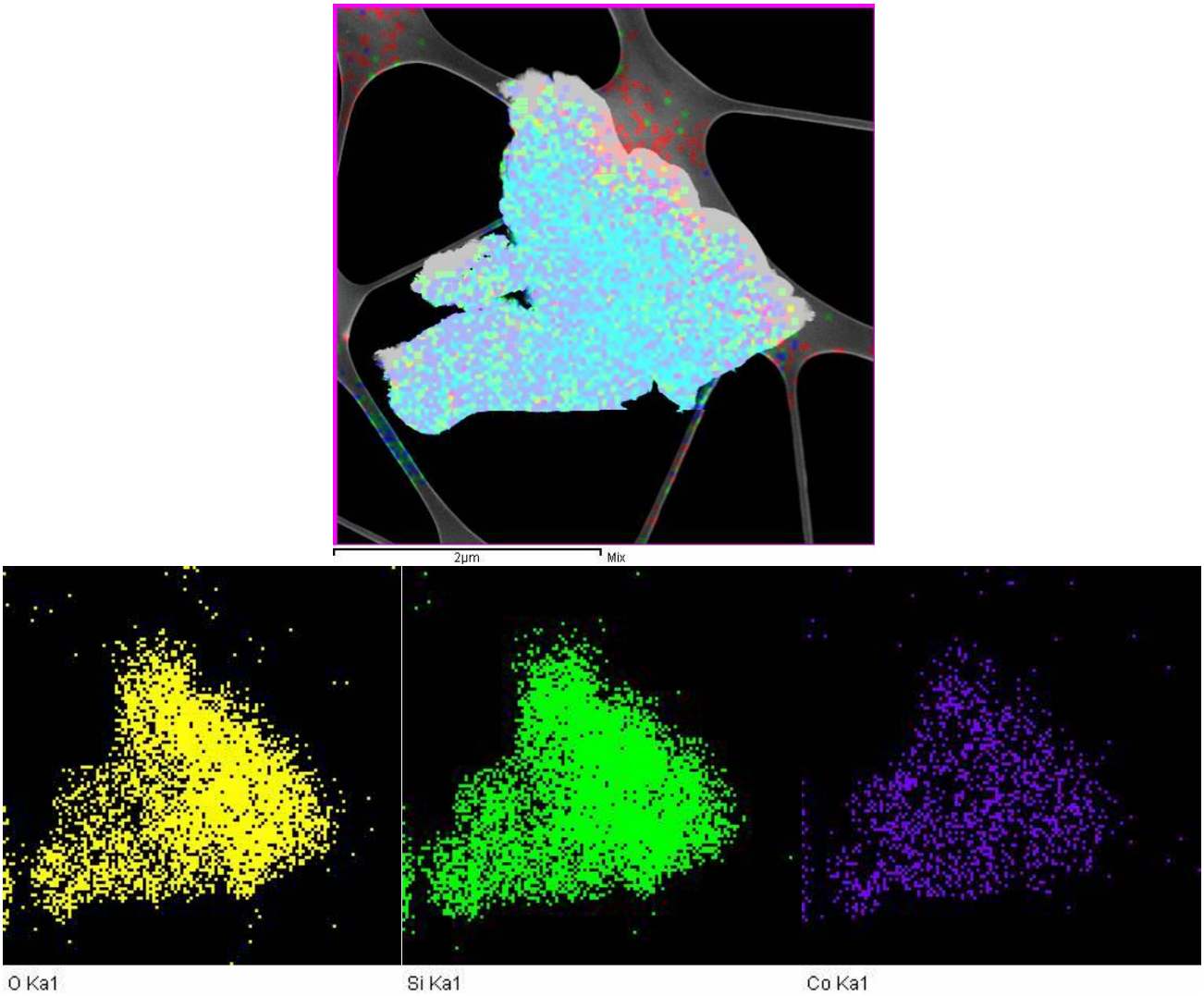
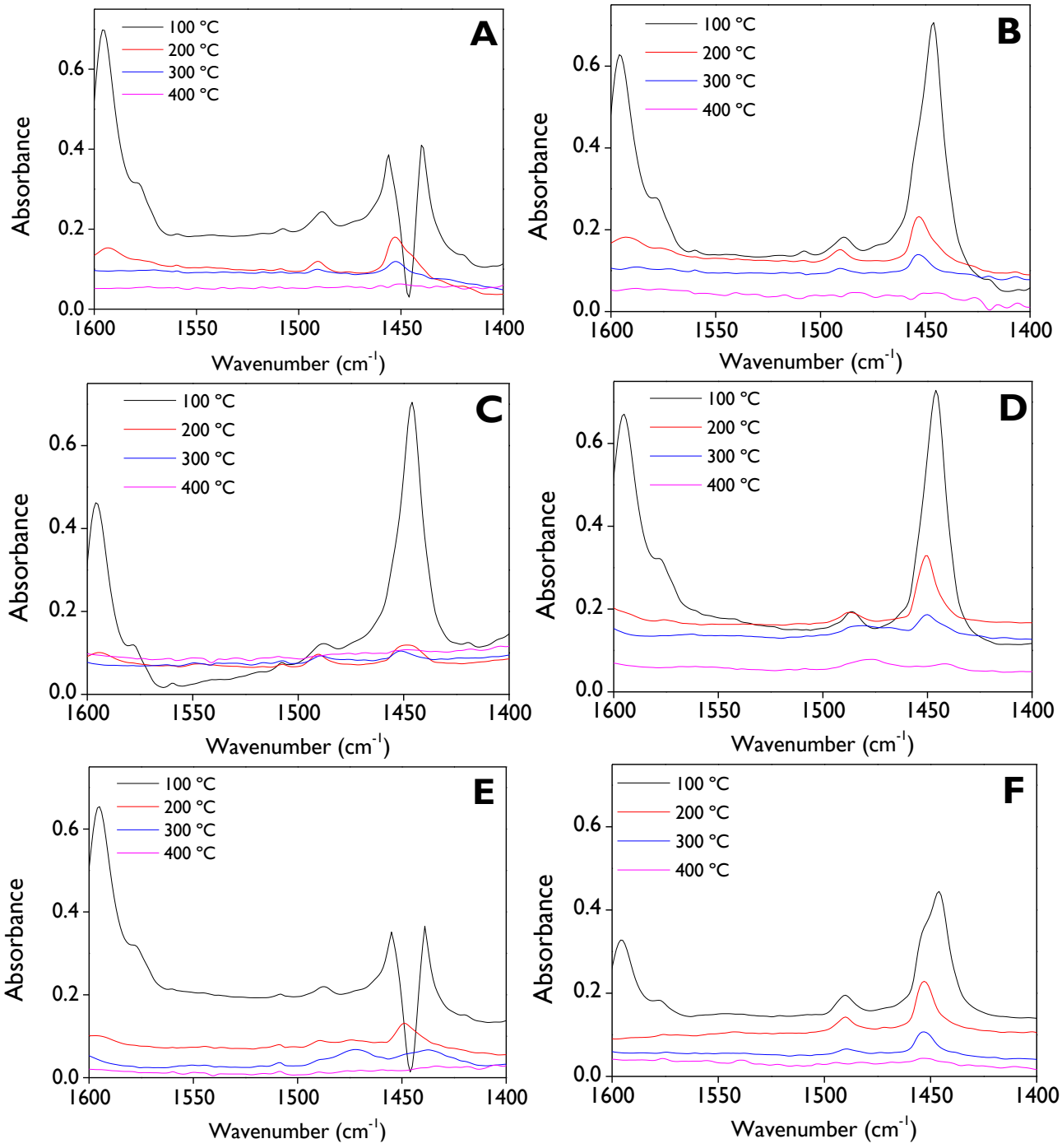


Figure S19. Elemental maps of the CoS1 catalyst.

4. Pyridine-FTIR



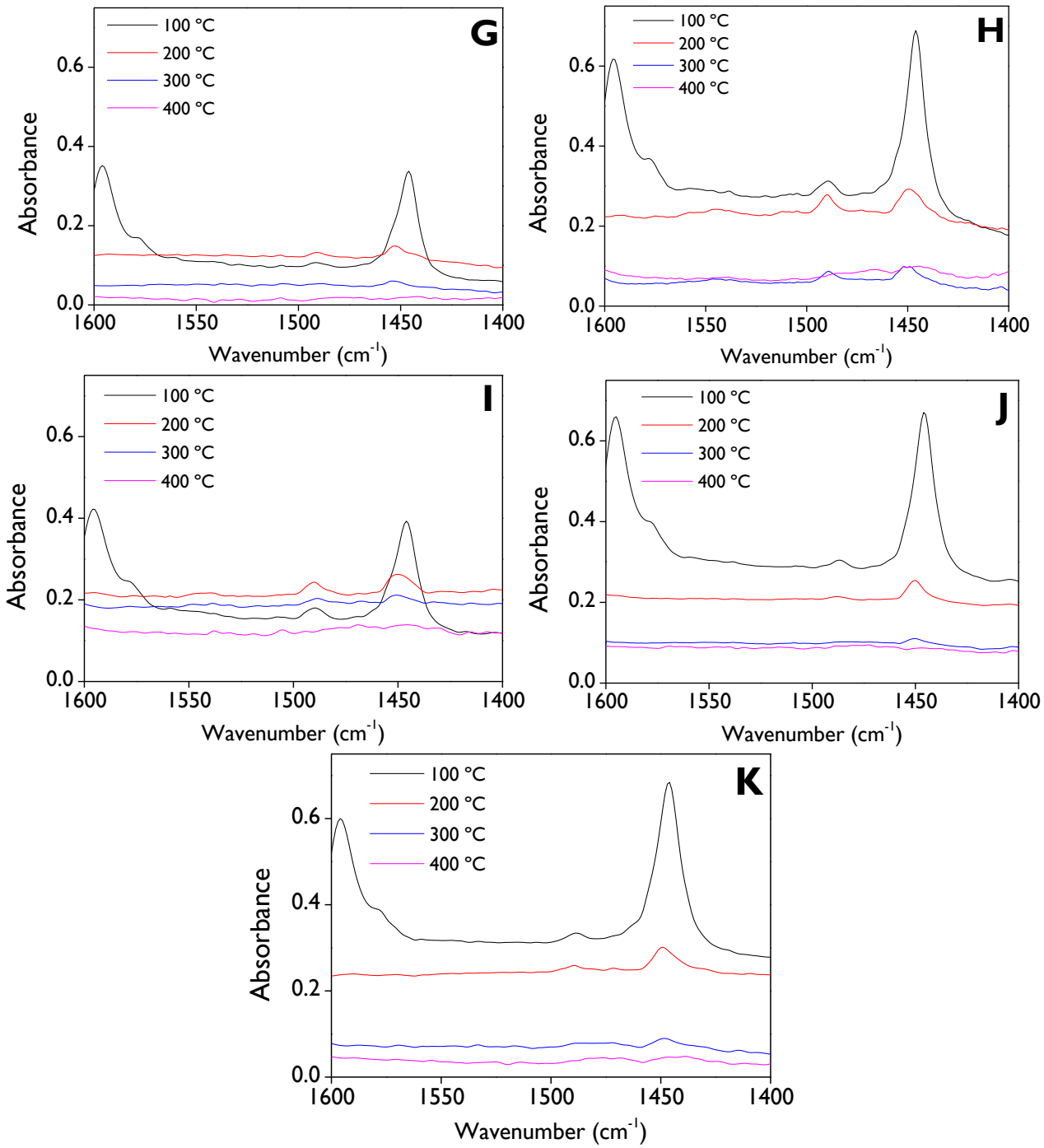


Figure S20. Pyridine-FTIR spectra of **(A)** SnM1, **(B)** SnM2, **(C)** FeM1, **(D)** CuM1, **(E)** CoM1, **(F)** SnS1, **(G)** SnS2, **(H)** FeS1, **(I)** FeS2, **(J)** CuS1, **(K)** CoS1.

5. NH_3-TPD Analysis

Table S1. Acidic properties of the catalysts using NH_3-TPD.

Catalyst	Acidity (μmol g^{-1})			
	Very weak	**Weak**	**Medium**	**Total**
SnM1	15	39	89	143
SnM2	13	48	81	141
FeM1	19	31	106	156
CuM1	16	63	180	259
CoM1	14	39	70	123
SnS1	20	54	90	164
SnS2	26	40	74	141
FeS1	49	0	146	195
FeS2	48	0	114	162
CuS1	0	67	92	159
CoS1	10	30	78	118

6. XPS Analysis

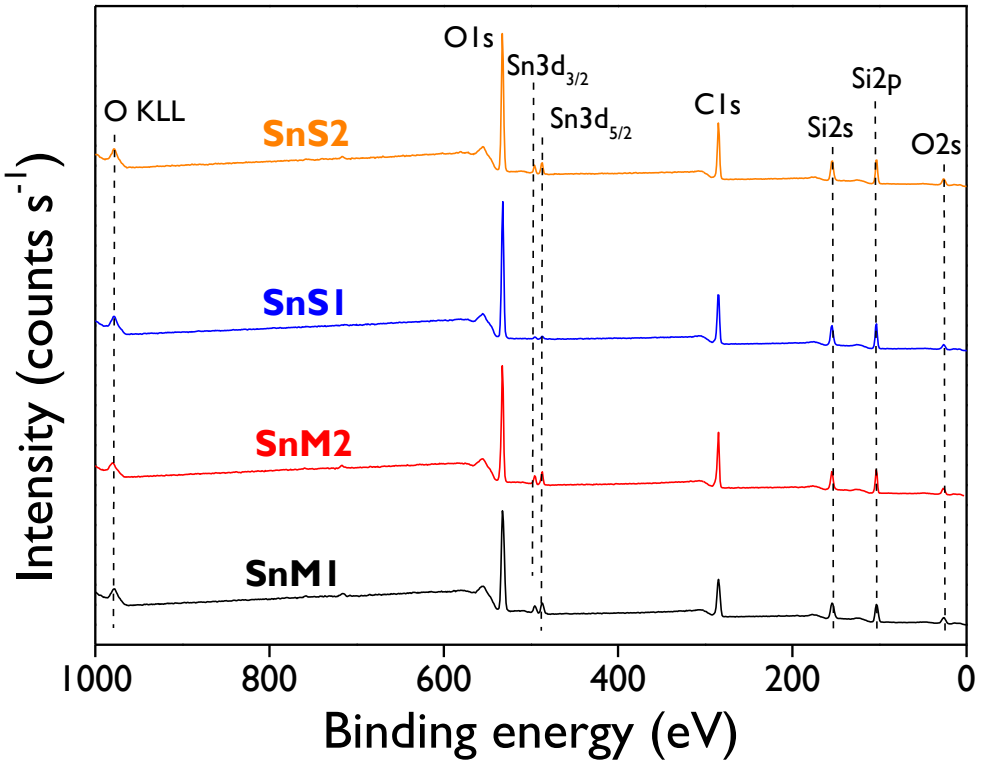


Figure S21. Wide scan XPS spectra of catalysts modified with Sn.

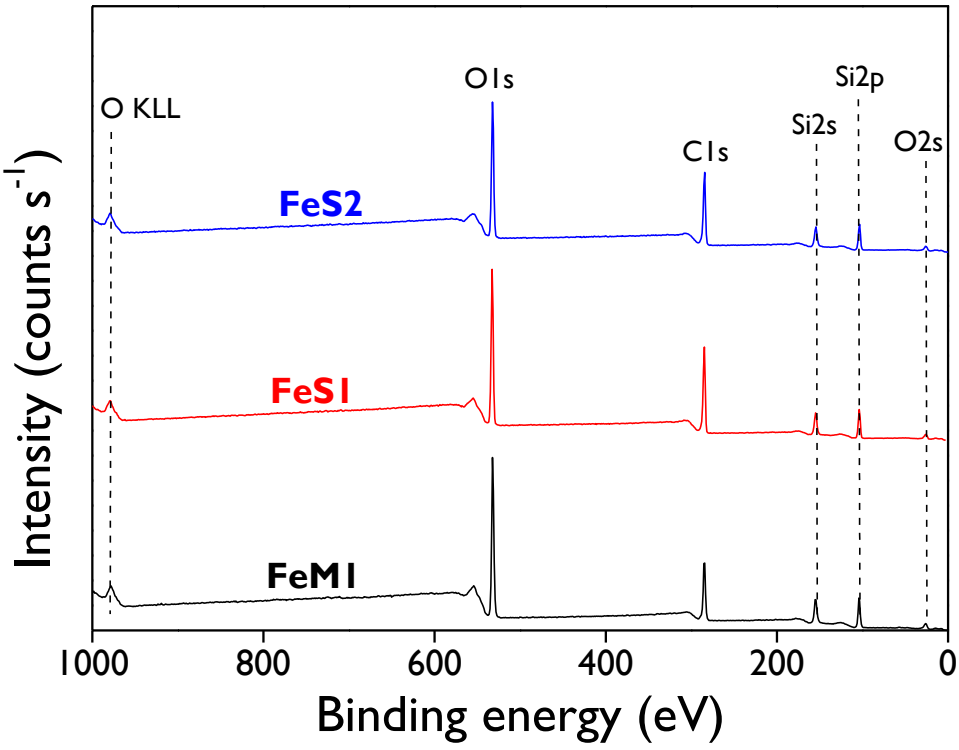


Figure S22. Wide scan XPS spectra of catalysts modified with Fe.

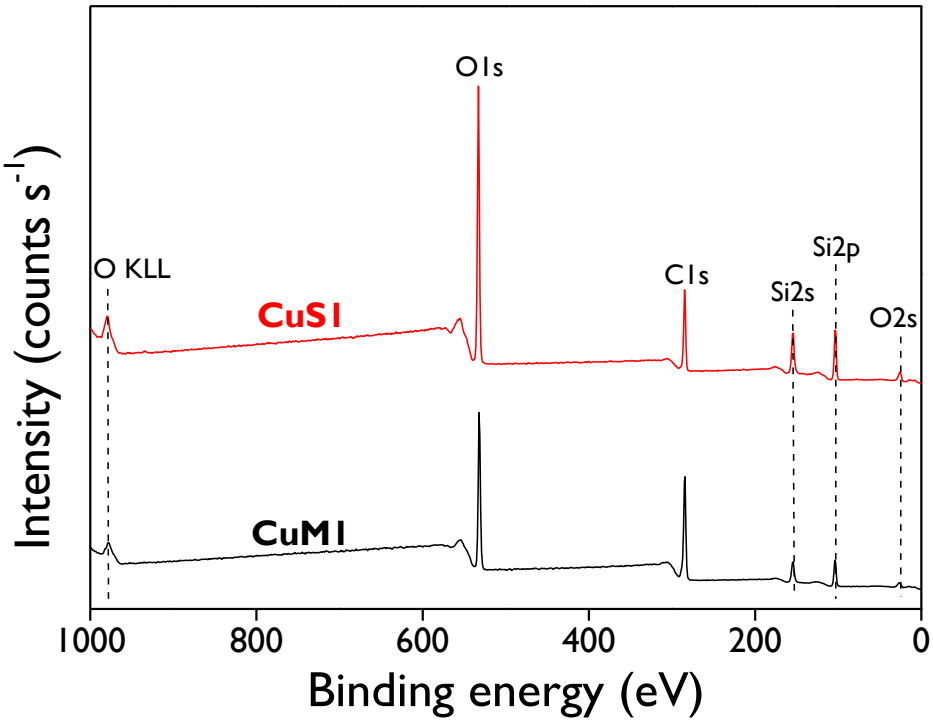


Figure S23. Wide scan XPS spectra of catalysts modified with Cu.

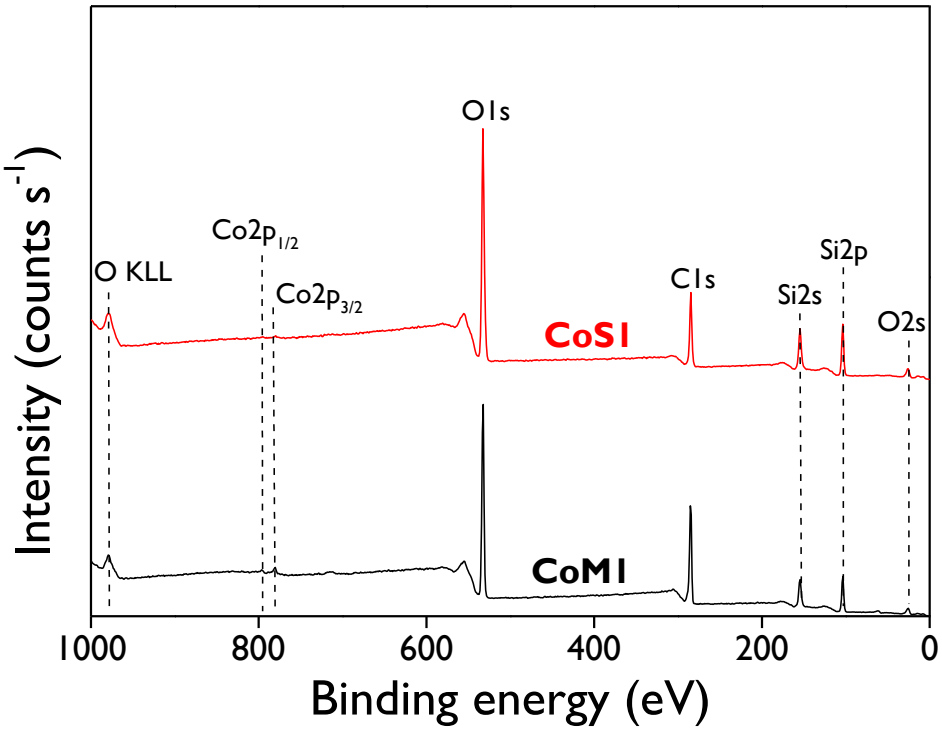


Figure S24. Wide scan XPS spectra of catalysts modified with Co.

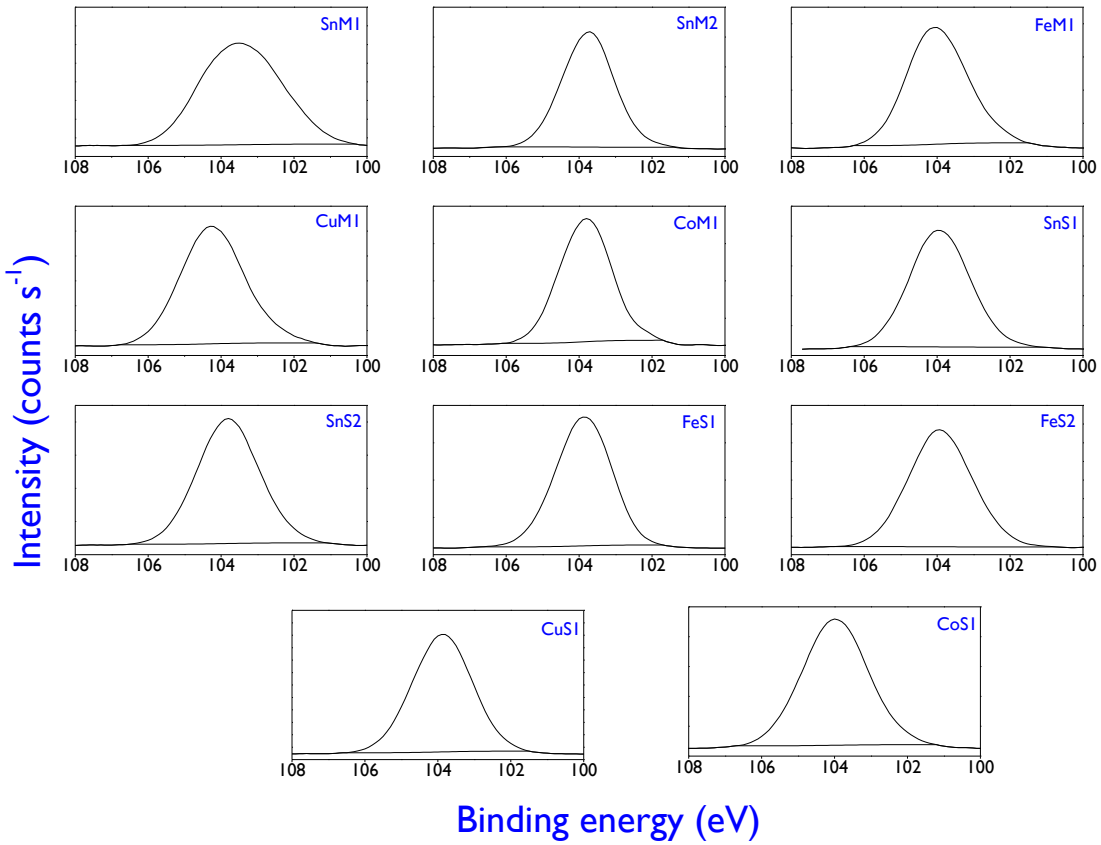


Figure S25. High-resolution XPS spectra for the Si2p region of all catalysts.

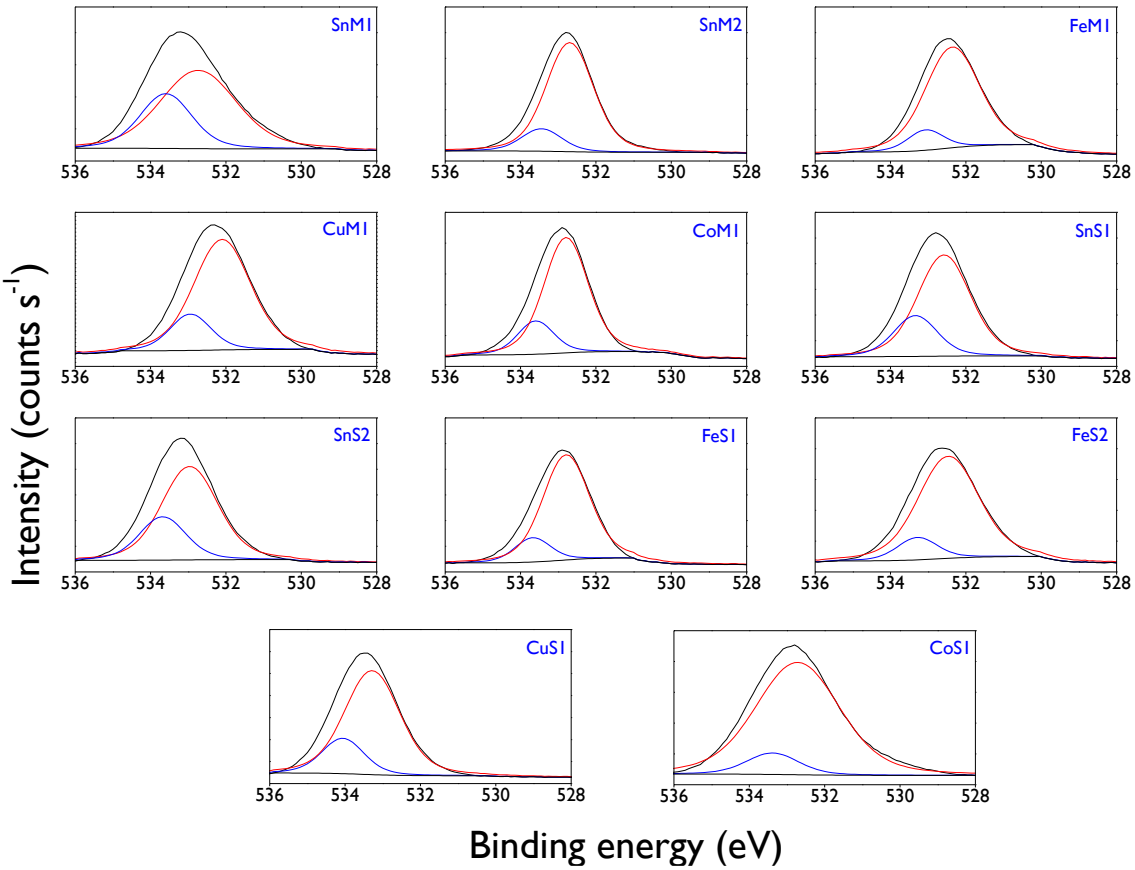


Figure S26. High-resolution XPS spectra for the O1s region of all catalysts.

7. Characterization of substrate and reaction products

Table S2. Retention times for the compounds involved in the one-pot transformation of β-pinene.

Compound	Retention time (min)	Chemical formula
β-Pinene (a)	3.95	$C_{10}H_{16}$
Product 1	5.60	$C_{10}H_{16}O$
Product 2	6.05	$C_{10}H_{16}O$
Product 3	6.23	$C_{10}H_{16}O$
β-Pinene epoxide (b)	6.45	$C_{10}H_{16}O$
Product 4	6.60	$C_{10}H_{16}O$
Product 5	6.67	$C_{10}H_{16}O$
Cis-Myrtanal (c_1)	6.76	$C_{10}H_{16}O$
Trans-Myrtanal (c_2)	6.85	$C_{10}H_{16}O$
Myrtenol (d)	7.20	$C_{10}H_{16}O$
Perillyl alcohol (e)	8.69	$C_{10}H_{16}O$
Product 6	9.94	$C_{10}H_{18}O_2$

Possible identification of the products:

Product 1: α-Pinene epoxide (CAS number: 72936-74-4) with quality GC-MS = 60%.
Product 2: Artemiseole (CAS number: 60485-46-3) with low-quality GC-MS = 28%.
Product 4: Bicyclo[2.2.1]heptane-2,5-diol, 1, 7, 7-trimethyl-, (2-endo, 5-exo)- (CAS number: 10359-41-8) with low-quality GC-MS = 40%.
Product 6: Bicyclo[3.1.0]hexane-6-methanol,2-hydroxy-1,4,4-trimethyl (CAS number: 58795-41-8) with low-quality GC-MS = 25%.

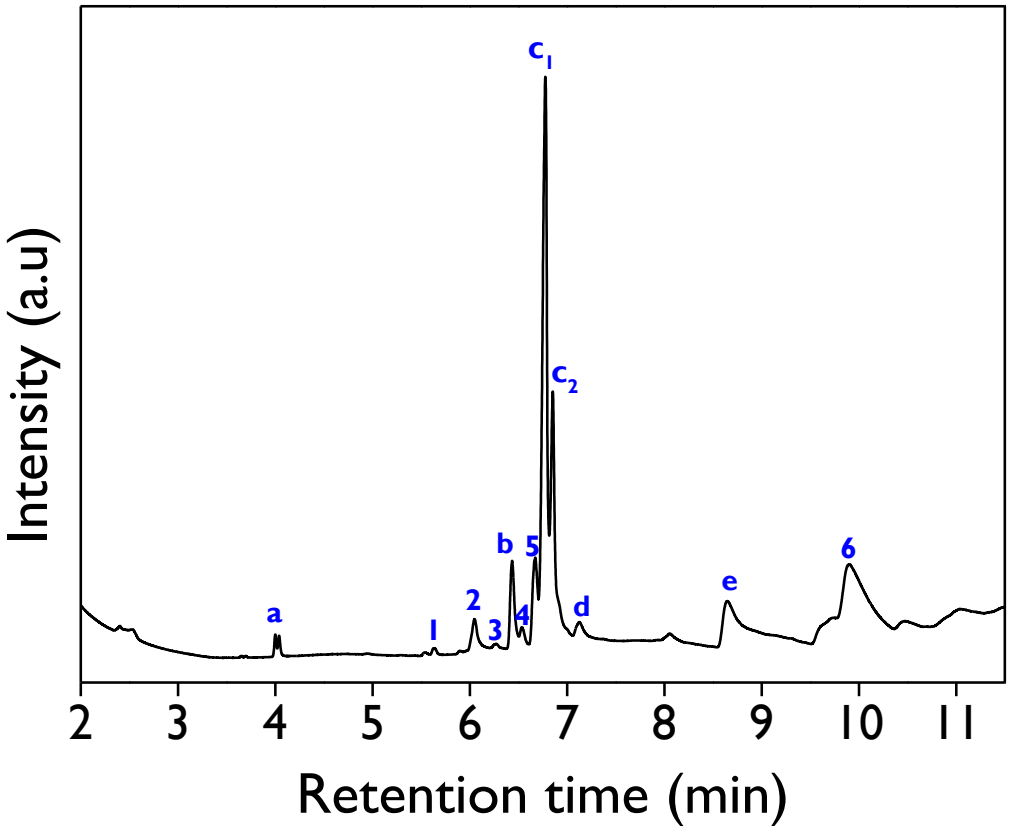


Figure S27. Representative chromatogram of a reaction sample after 48 h in the one-pot transformation of β-pinene. **Reaction conditions:** 0.10 mmol β-pinene, 1: 1.2: 0.72: 30.3: 19.7: 0.8: 15.7 as weight ratios for β-pinene: MgO: acid catalyst: H_2O: acetone: H_2O_2: acetonitrile, 50 °C, 1000 rpm.

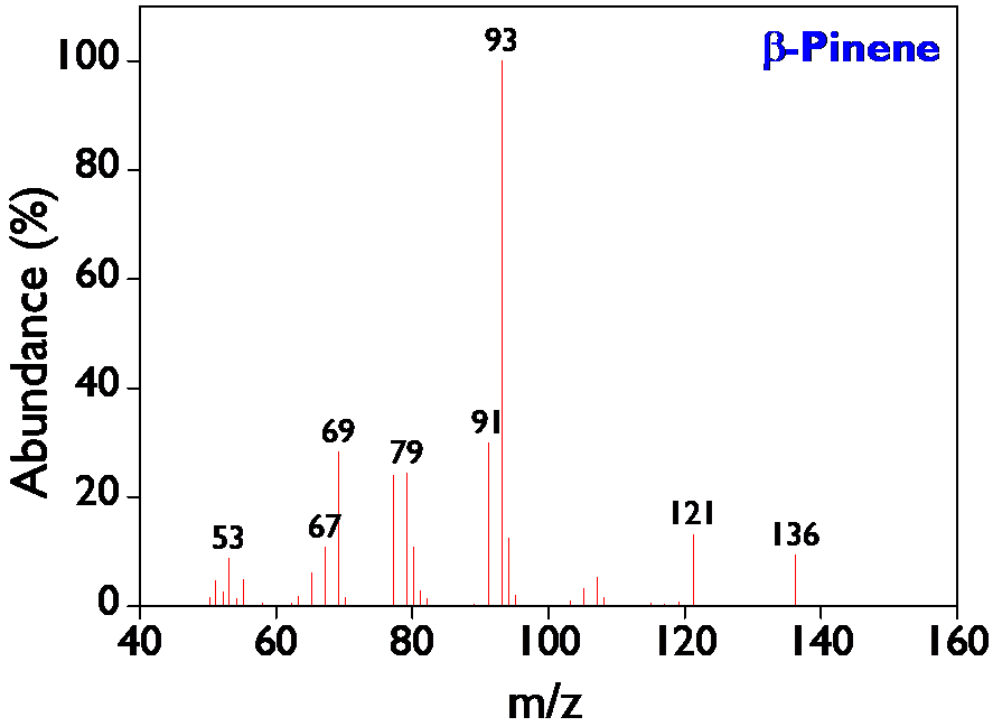


Figure S28. Mass spectrum of the β-pinene.

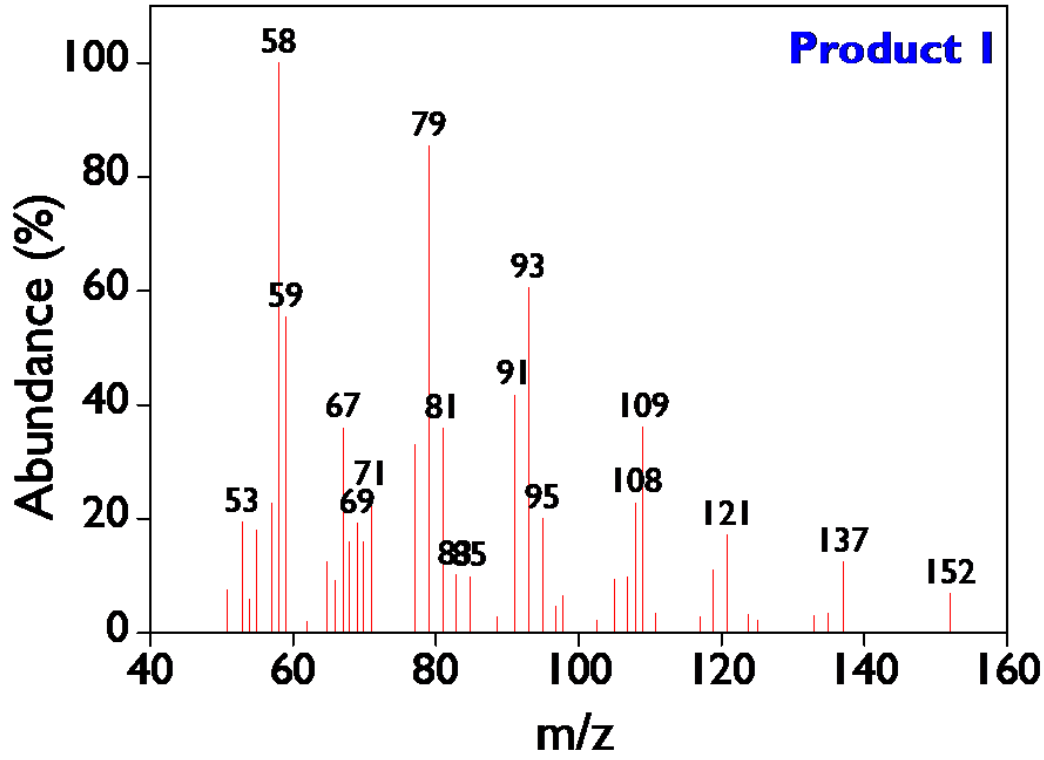


Figure S29. Mass spectrum of the product 1.

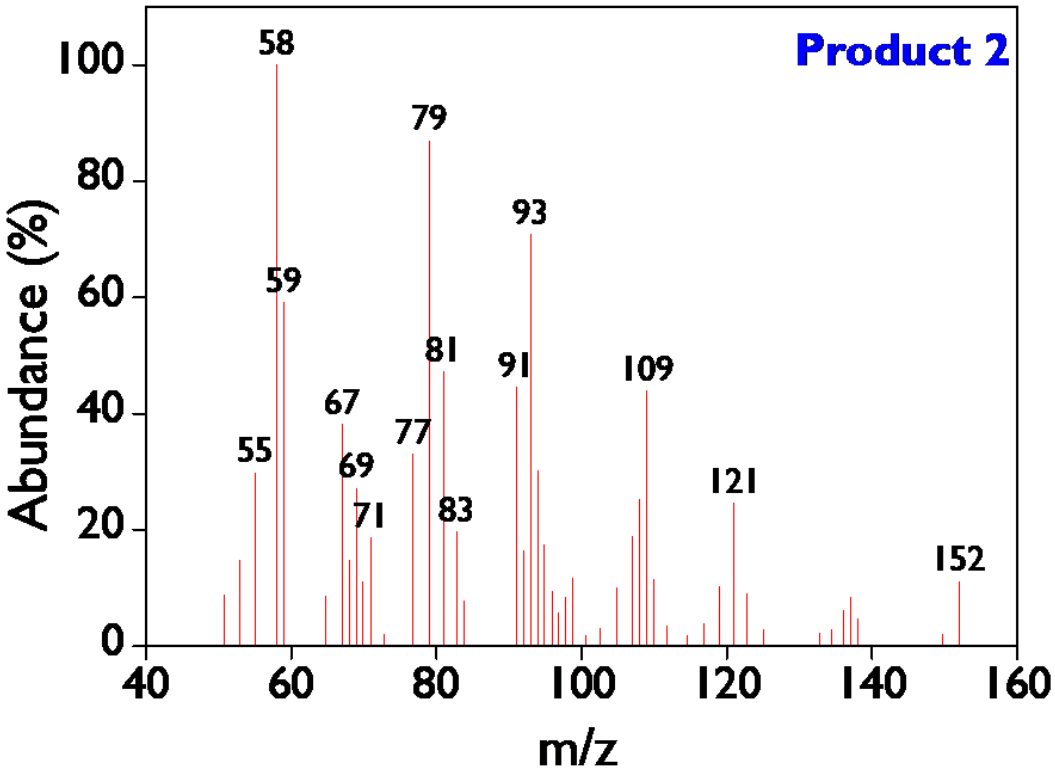


Figure S30. Mass spectrum of the product 2.

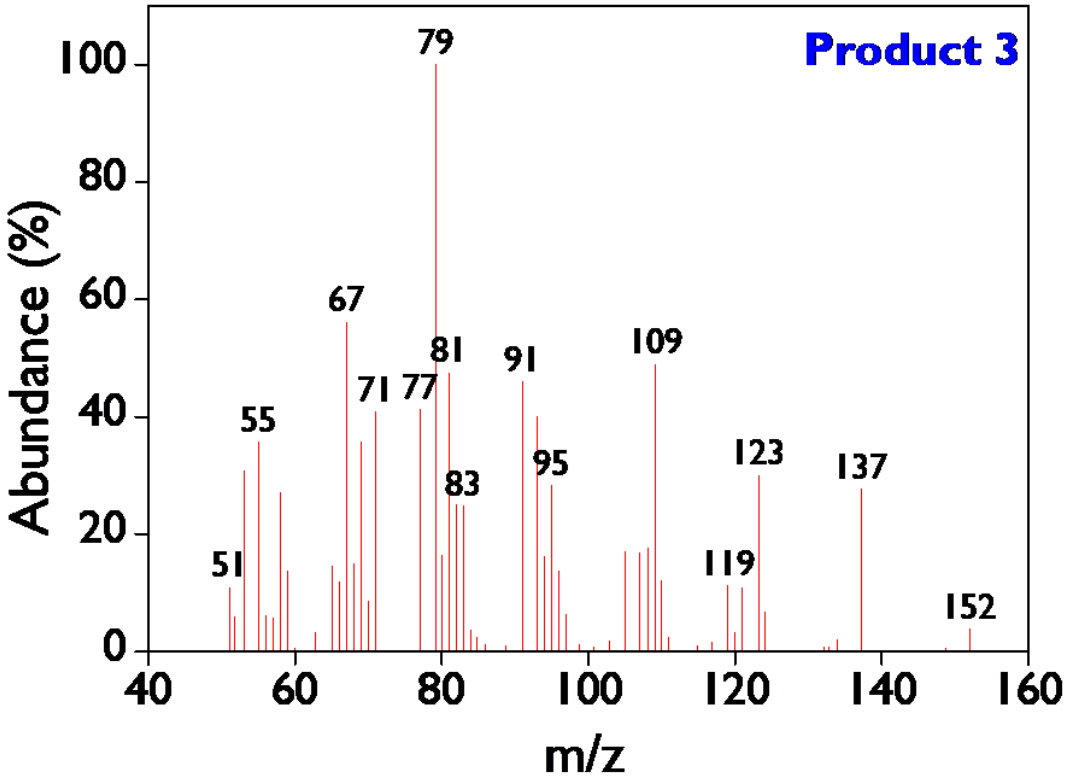


Figure S31. Mass spectrum of the product 3.

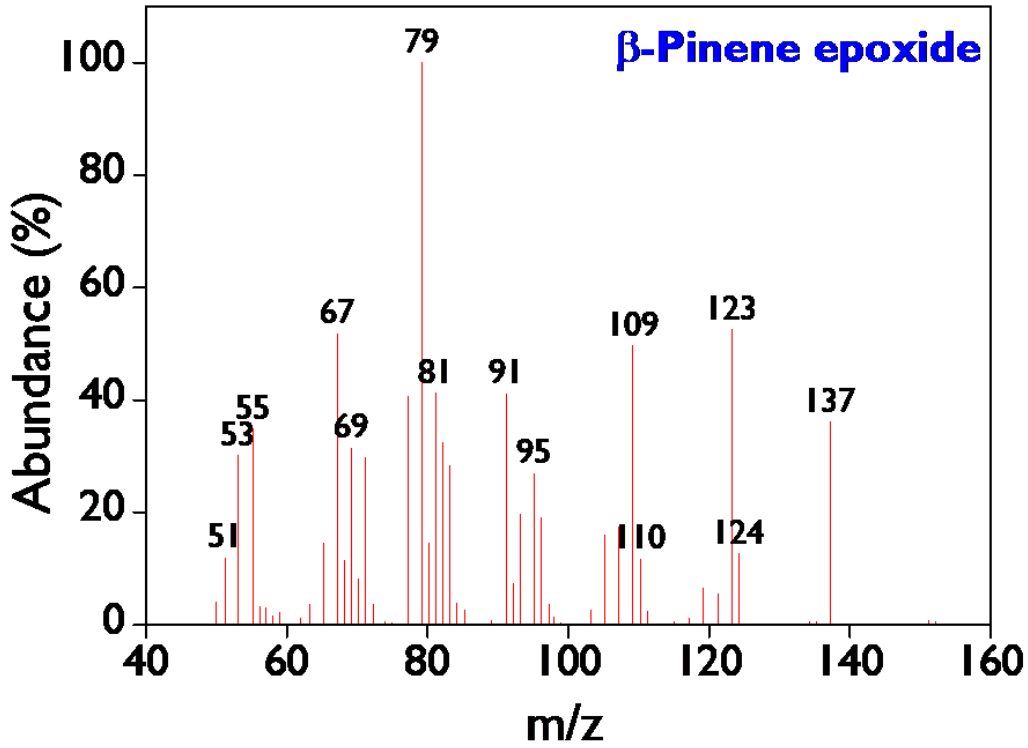


Figure S32. Mass spectrum of the β-pinene epoxide.

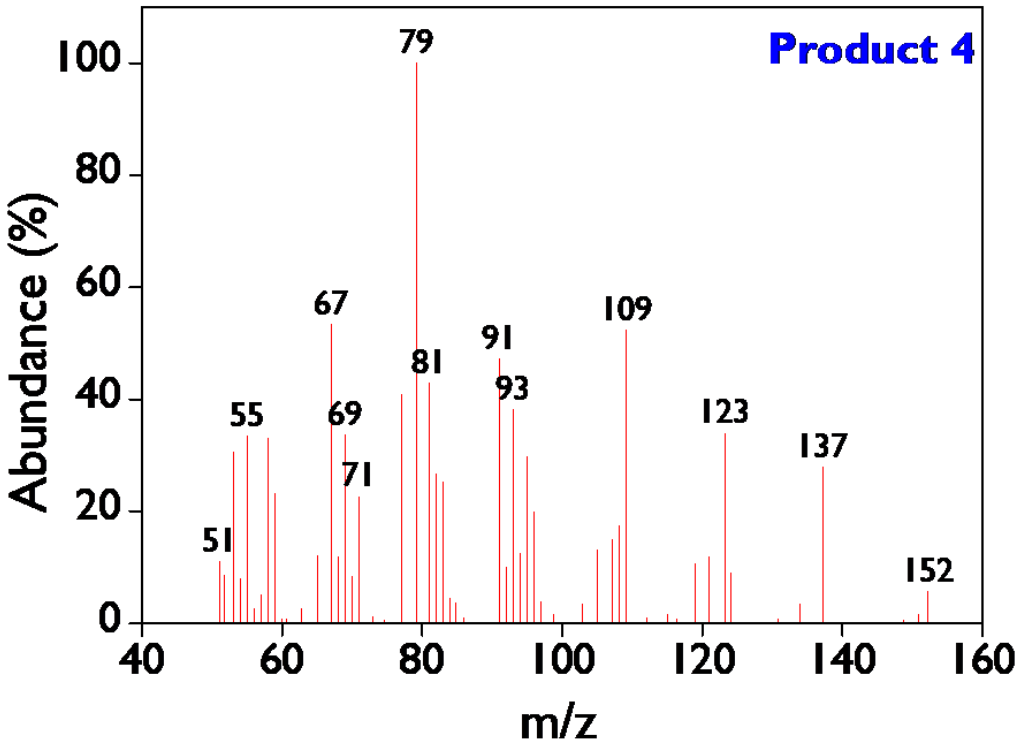


Figure S33. Mass spectrum of the product 4.

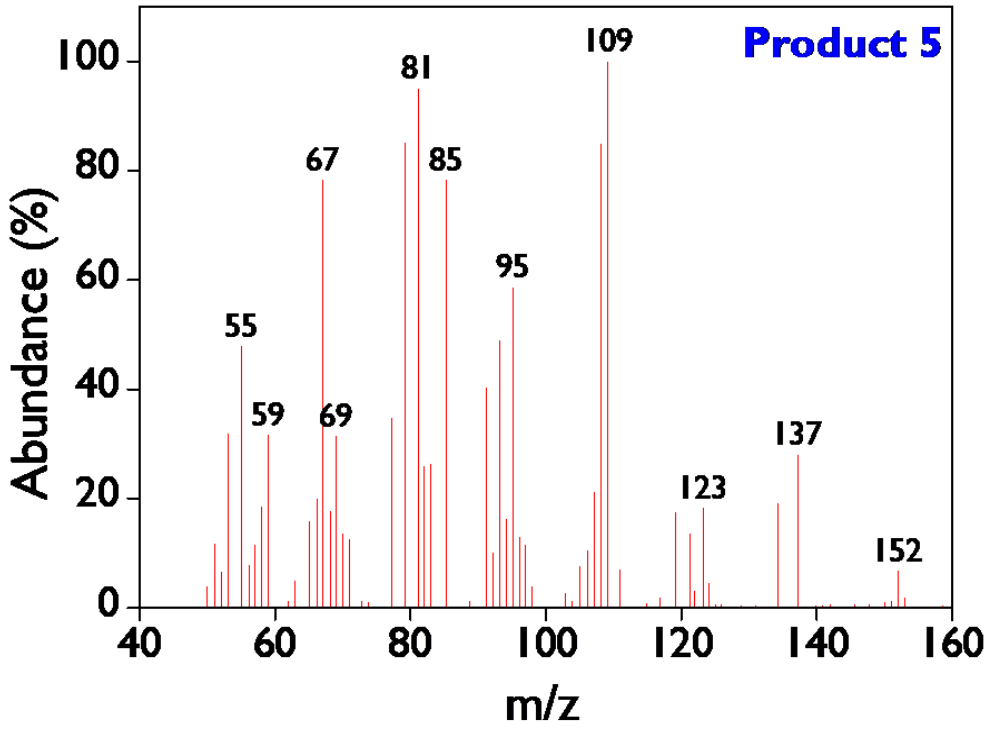


Figure S34. Mass spectrum of the product 5.

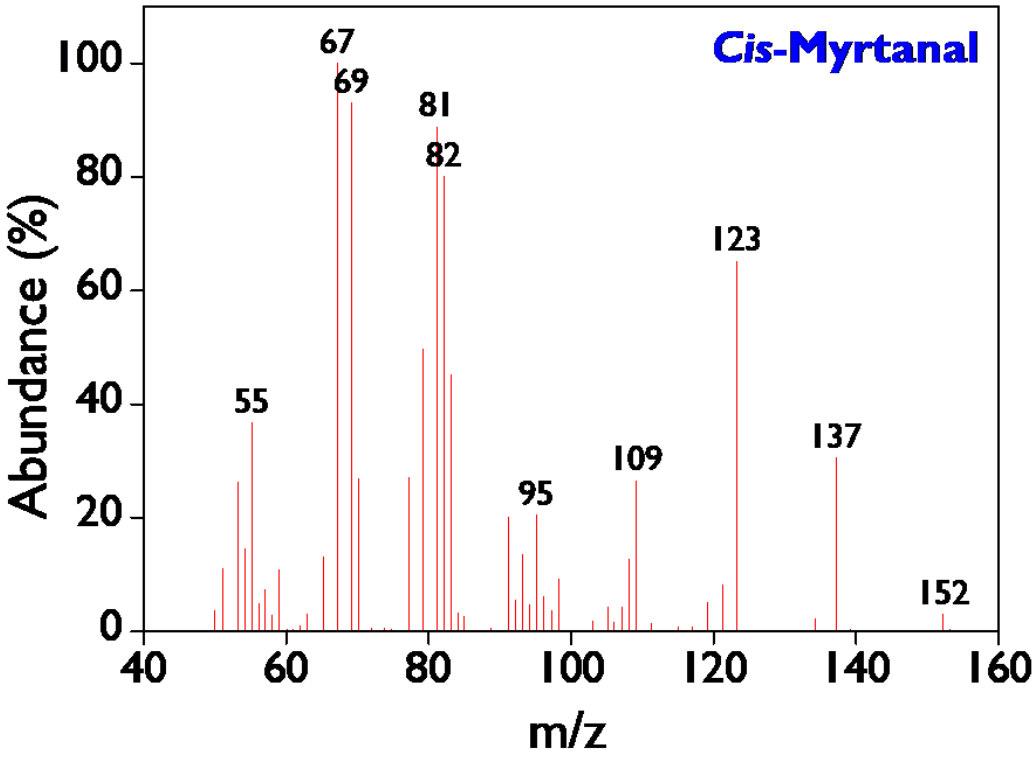


Figure S35. Mass spectrum of the *cis*-myrtanal.

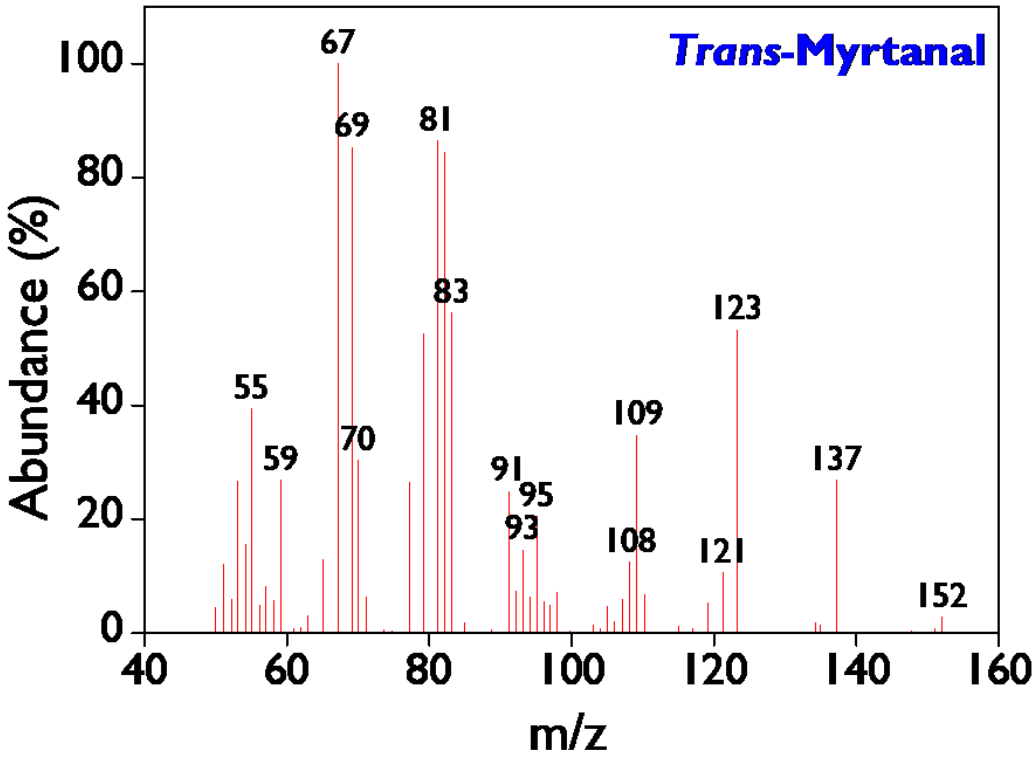


Figure S36. Mass spectrum of the *trans*-myrtanal.

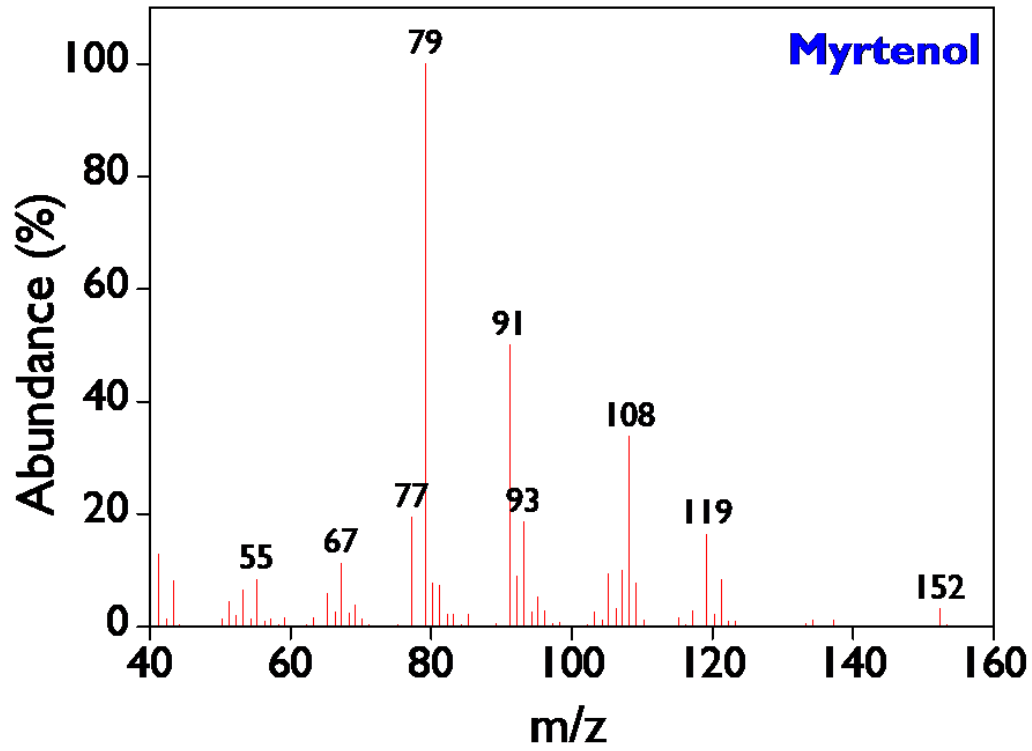


Figure S37. Mass spectrum of the myrtenol.

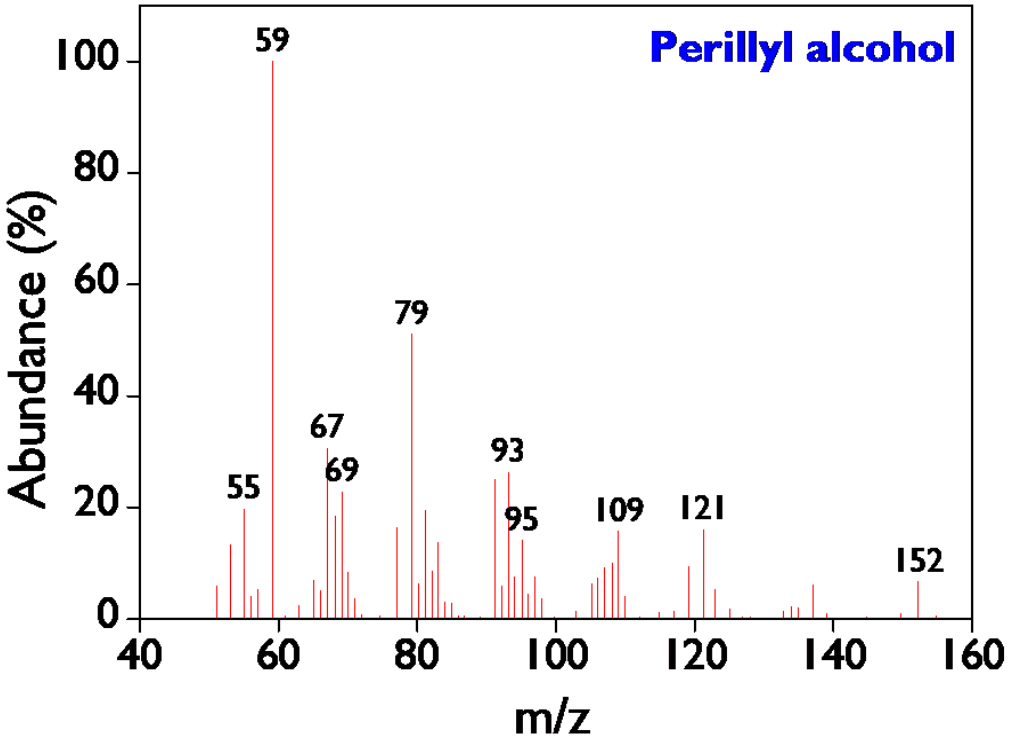


Figure S38. Mass spectrum of the perillyl alcohol.

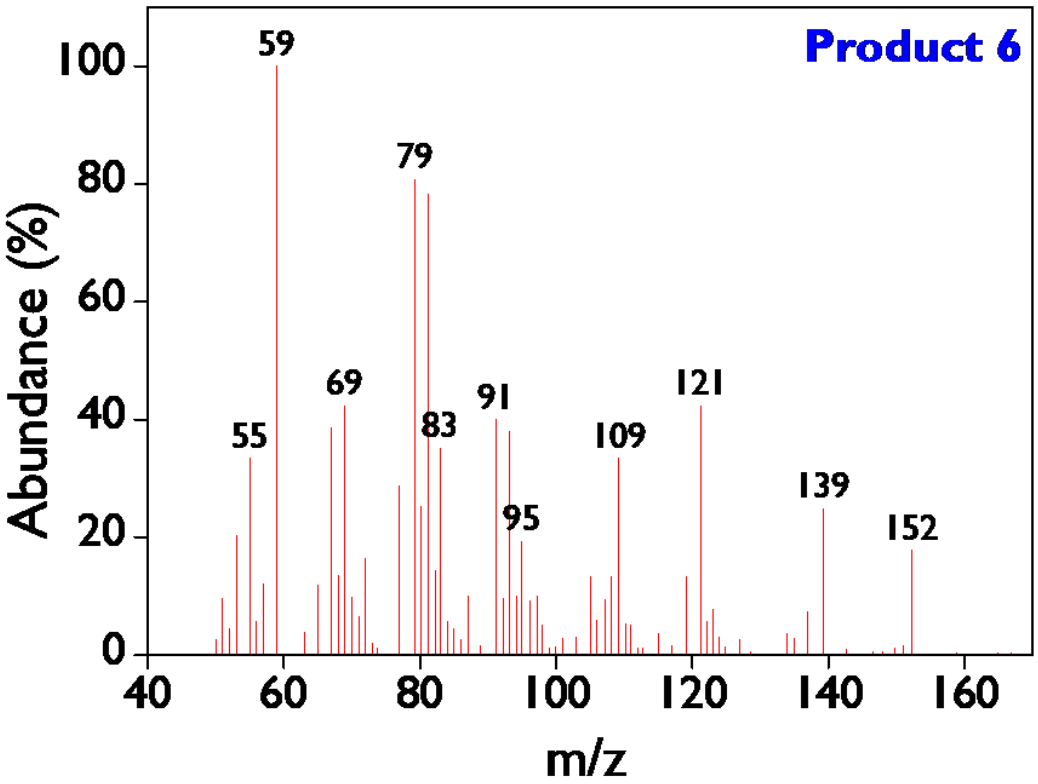


Figure S39. Mass spectrum of the product 6.

8. Repeatability test

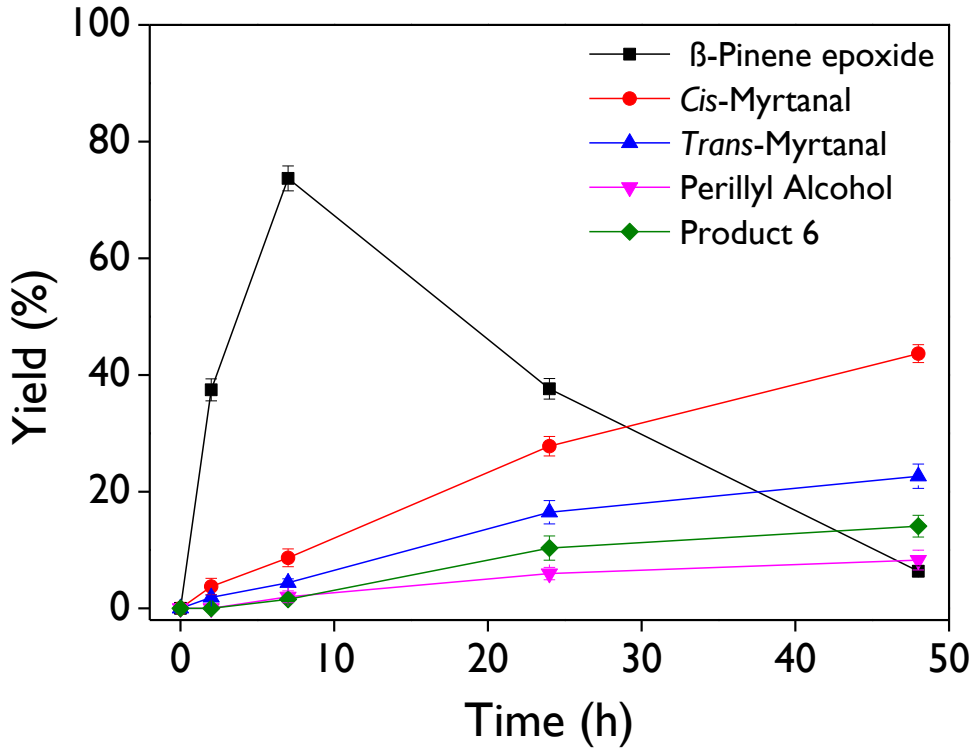


Figure S40. Repeatability test in the one-pot transformation of β-pinene over FeS1 as a catalyst. **Reaction conditions:** 0.1 mmol of β-pinene with weight ratios of 1: 0.72: 1.2: 30.3: 19.7: 15.7: 0.8 for β-pinene: acidic catalyst: MgO: H_2O: acetone: acetonitrile: H_2O_2, 50 °C, 1000 rpm. Figures were constructed with error bars (standard deviation), which were obtained from three experimental runs under the same conditions.

9. Role of the acidic catalyst

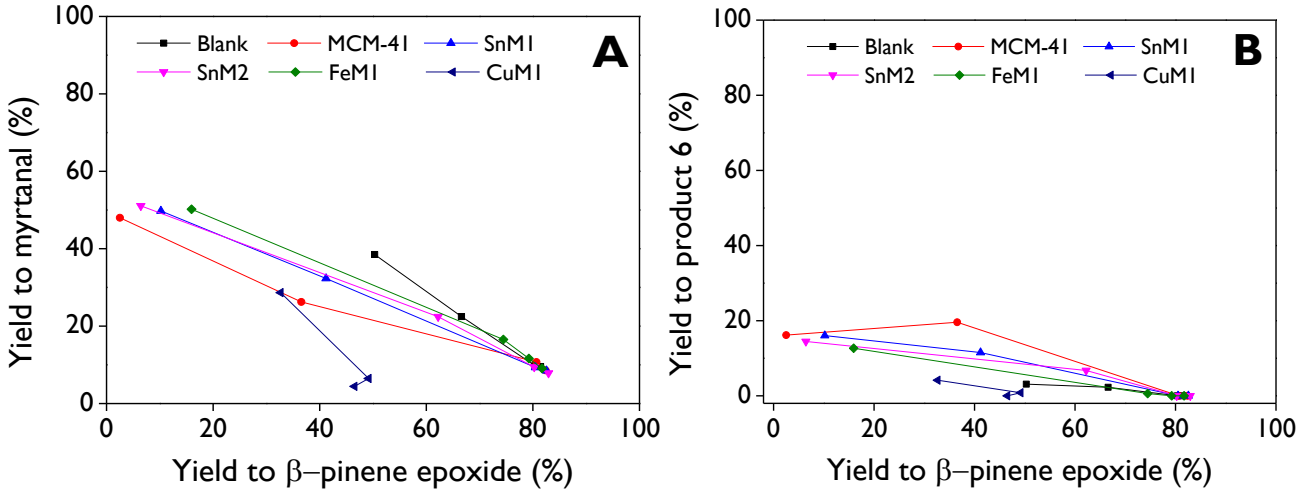


Figure S41. Role of acidic catalysts based on MCM-41 support in the one-pot transformation of β-pinene: **(A)** yield of myrtanal vs. yield of β-pinene epoxide, and **(B)** yield of product 6 vs. yield of β-pinene epoxide. **Reaction conditions:** 0.1 mmol of β-pinene with weight ratios of 1: 0.72: 1.2: 30.3: 19.7: 15.7: 0.8 for β-pinene: acidic catalyst: MgO: H_2O: acetone: acetonitrile: H_2O_2, 50 °C, 1000 rpm.

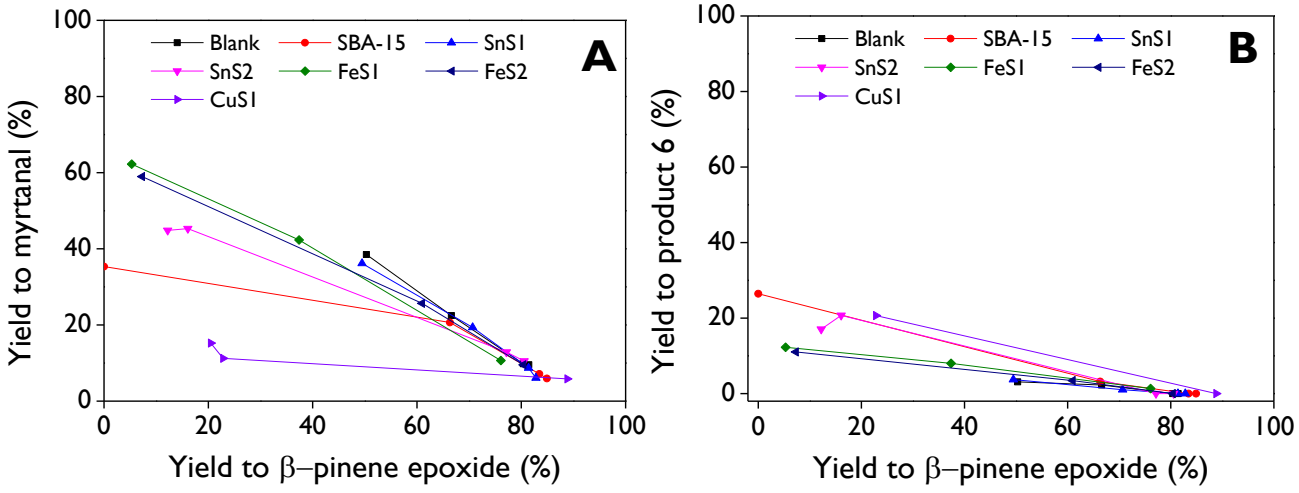


Figure S42. Role of acidic catalysts based on SBA-15 support in the one-pot transformation of β-pinene: **(A)** yield of myrtanal vs. yield of β-pinene epoxide, and **(B)** yield of product 6 vs. yield of β-pinene epoxide. **Reaction conditions:** 0.1 mmol of β-pinene with weight ratios of 1: 0.72: 1.2: 30.3: 19.7: 15.7: 0.8 for β-pinene: acidic catalyst: MgO: H_2O: acetone: acetonitrile: H_2O_2, 50 °C, 1000 rpm.

10. Conversion profiles of H_2O_2

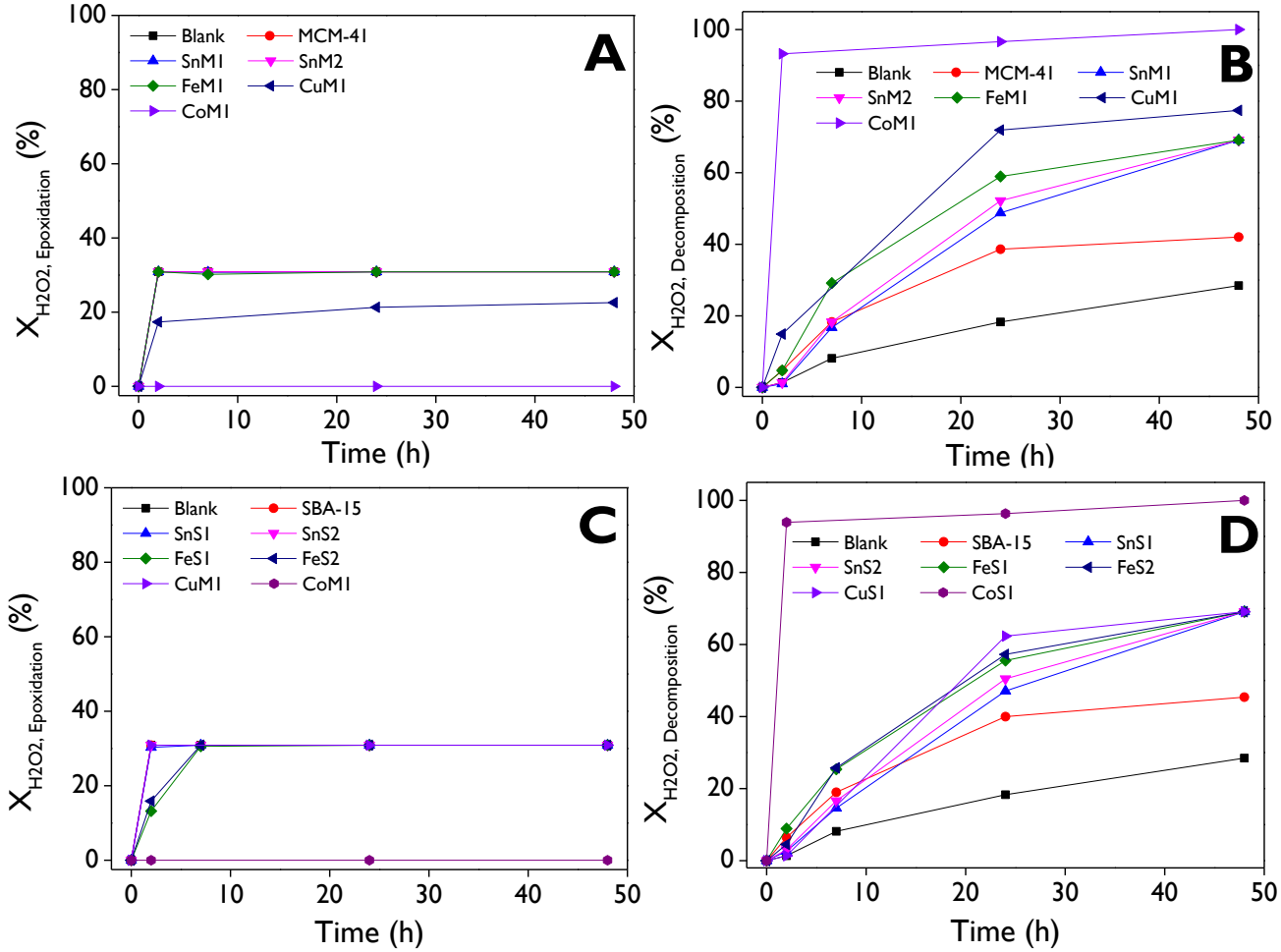


Figure S43. Profiles of the H_2O_2 conversion into the epoxidation reaction (**A, C**) and the decomposition route **(B, D)** with the catalysts based on MCM-41 **(A, B)** and SBA-15 **(C, D)**. **Reaction conditions:** 0.1 mmol of β-pinene with weight ratios of 1: 0.72: 1.2: 30.3: 19.7: 15.7: 0.8 for β-pinene: acidic catalyst: MgO: H_2O: acetone: acetonitrile: H_2O_2, 50 °C, 1000 rpm. Calculations were performed based on a recent contribution [1].

11. Effect of the reaction conditions

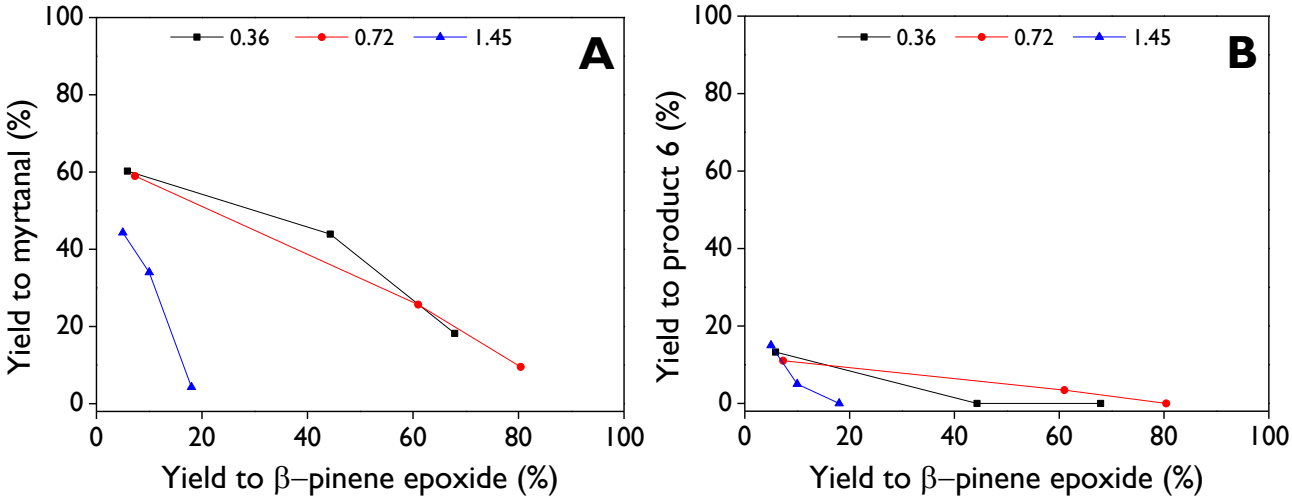


Figure S44. Effect of amount of FeS2 in the one-pot transformation of β-pinene: **(A)** yield of myrtanal vs. yield of β-pinene epoxide, and **(B)** yield of product 6 vs. yield of β-pinene epoxide. **Reaction conditions:** 0.1 mmol of β-pinene with weight ratios of 1: (0.36, 0.72, 1.45): 1.2: 30.3: 19.7: 15.7: 0.8 for β-pinene: acidic catalyst: MgO: H_2O: acetone: acetonitrile: H_2O_2, 50 °C, 1000 rpm.

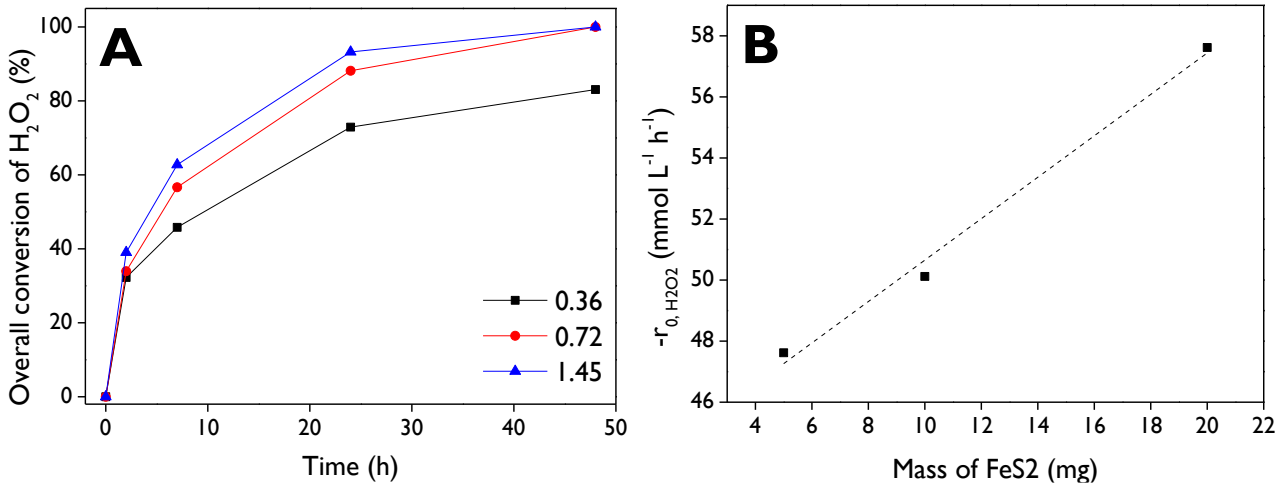


Figure S45. Effect of amount of FeS2 in the one-pot transformation of β-pinene: **(A)** Global conversion of H_2O_2, and **(B)** initial reaction rate of H2O2 as a function of mass of FeS2. **Reaction conditions:** 0.1 mmol of β-pinene with weight ratios of 1: (0.36, 0.72, 1.45): 1.2: 30.3: 19.7: 15.7: 0.8 for β-pinene: acidic catalyst: MgO: H_2O: acetone: acetonitrile: H_2O_2, 50 °C, 1000 rpm.

12. References

[1] L.A. Gallego-Villada, P. Mäki-Arvela, N. Kumar, E.A. Alarcón, Z. Vajglová, T. Tirri, I. Angervo, R. Lassfolk, M. Lastusaari, D.Y. Murzin, Zeolite Y-based catalysts for efficient epoxidation of R-(+)-Limonene: Insights into the structure-activity relationship, Microporous Mesoporous Mater. 372 (2024) 113098. https://doi.org/10.1016/j.micromeso.2024.113098.