Supplementary information:

Structural, morphological and optical investigation of β-Ag_2MoO_4 microcrystals obtained with different polar solvents

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Experimental details

β-Ag_2MoO_4 microcrystals were synthesized as follows: 1×10^{-3} mols of molybdate sodium dihydrate ($Na_2MoO_4.2H_2O$; 99.5% purity, Sigma-Aldrich) and 2×10^{-3} mols of silver acetate (CH_3COOAg; 99.99% purity, Sigma-Aldrich) were dissolved in 200 mL of five polar solvents: deionized water (H_2O), methanol (CH_4O), ethanol (C_2H_6O), 1-propanol (C_3H_8O) and 1-butanol ($C_4H_{10}O$) at 60°C for 8 h. In order to minimize the evaporation losses, the volume in each synthesis was maintained constant (200 mL) with the addition of the respective solvent in the solution. Finished all precipitation reactions, the final products were washed and centrifuged with deionized water/acetone several times to remove any remaining Na^+ ion. Afterwards, the precipitates were dried at room temperature for 12 h, and then, collected.

Characterizations

The microcrystals were characterized by XRD using a D/Max-2500PC diffractometer (Rigaku, Japan) with Cu-Kα radiation (λ = 1.5406 Å) in the 2θ range from 10° to 70° with a scanning velocity of 2° min^{-1} in the normal routine, and from 10° to 110° with a scanning velocity of 1° min^{-1} in the Rietveld routine. The crystal shapes and sizes were observed with a Supra 35-VP field-emission scanning electron microscope (Carl Zeiss, Germany), operated at 15 kV. UV–vis spectra were recorded using a Cary 5G spectrophotometer (Varian, USA) in diffuse reflectance mode.

Figures:

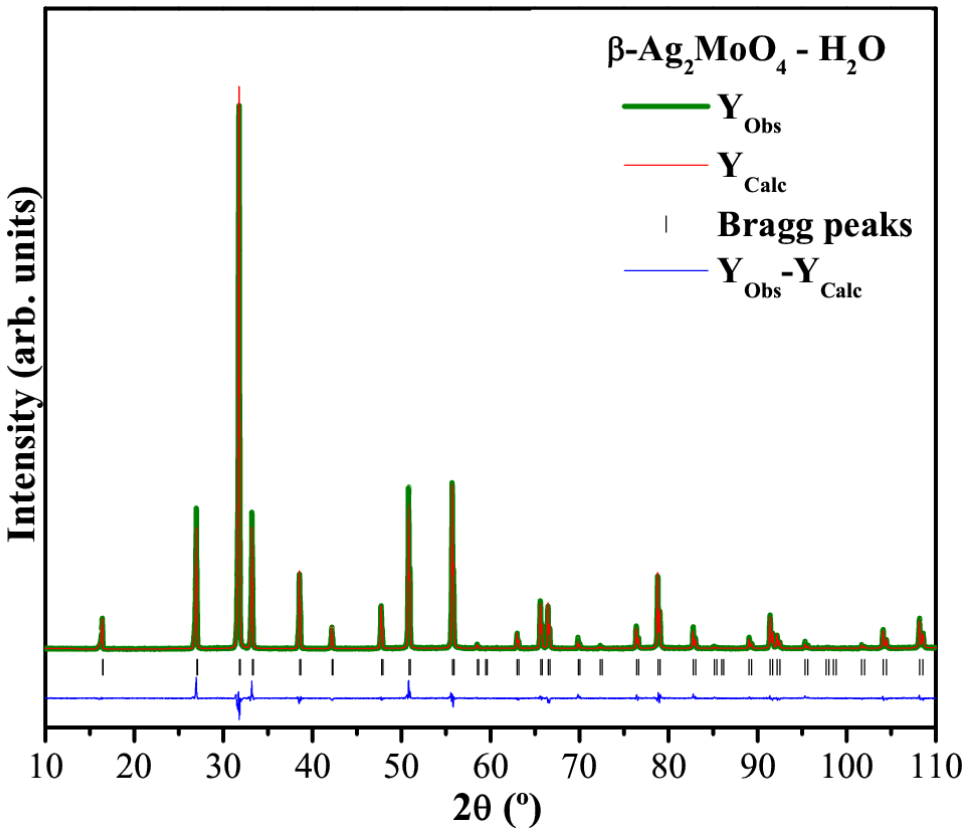

Fig. S1: Rietveld refinement plot of β-Ag_2MoO_4 microcrystals synthesized at 60°C for 8 h with H_2O.

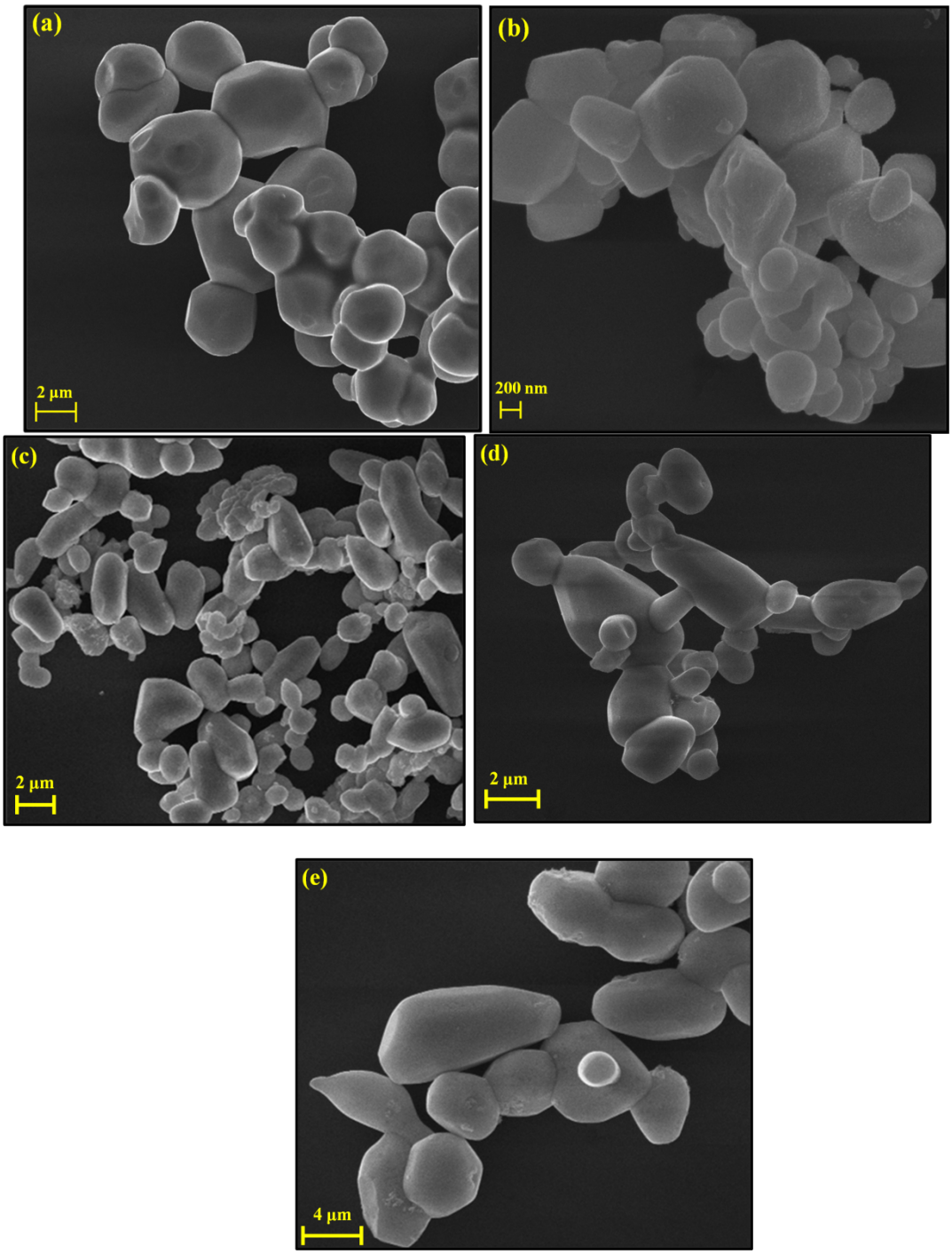

Fig. S2. FE-SEM images of β-Ag_2MoO_4 microcrystals synthesized at 60°C for 8 h with **(a)** H_2O, **(b)** CH_4O, **(c)** C_2H_6O, **(d)** C_3H_8O, and **(e)** $C_4H_{10}O$.

Tables:

Table S1. Data obtained from Rietveld refinements for β-Ag_2MoO_4 microcrystals prepared with different polar solvents

Solvents	**Chemical formula**	**Dipole moment (*C.m*)**	**Bond angles [O–Ag–O] (°)**	**Bond angles [O–Mo–O] (°)**
Water	H_2O	1.85	ρ (82.36)/φ (97.64)	ω (109.47)
Methanol	CH_4O	1.70	ρ (82.30)/φ (97.70)	ω (109.47)
Ethanol	C_2H_6O	1.69	ρ (82.10)/φ (97.90)	ω (109.47)
1-Propanol	C_3H_8O	1.65	ρ (82.23)/φ (97.77)	ω (109.47)
1-Butanol	$C_4H_{10}O$	1.52	ρ (82.12)/φ (97.88)	ω (109.47)

Table S2. Lattice parameters, unit cell volume and site occupancy obtained from Rietveld refinements of β-Ag_2MoO_4 microcrystals prepared with different polar solvents.

■Atoms	Wyckoff	Site	*x*	*y*	*z*	U [Å²]
Ag	16*d*	.-3m	0.50000	0.50000	0.50000	0.0173
Mo	8*a*	-43m	0.12500	0.12500	0.12500	0.0067
O	32*e*	.3m	0.23132	0.23132	0.23132	0.0353
[α = β = γ = 90º; *a* = *b* = *c* = 9.3185(3) Å and V = 809.17(0)Å³]; R_{wp} = 12.95%, R_p = 9.28%, R_{Bragg} = 6.75%, χ^2 = 1.31, *S* =1.14						
●Atoms	Wyckoff	Site	*x*	*y*	*z*	U [Å²]
Ag	16*d*	.-3m	0.50000	0.50000	0.50000	0.0228
Mo	8*a*	-43m	0.12500	0.12500	0.12500	0.0124
O	32*e*	.3m	0.23113	0.23113	0.23113	0.0219
[α = β = γ = 90º; *a* = *b* = *c* = 9.3183(6) Å and V = 809.11(1)Å³]; R_{wp} = 11.44%, R_p = 7.5%, R_{Bragg} = 4.42%, χ^2 = 1.29, *S* =1.13						
♦Atoms	Wyckoff	Site	*x*	*y*	*z*	U [Å²]
Ag	16*d*	.-3m	0.50000	0.50000	0.50000	0.0216
Mo	8*a*	-43m	0.12500	0.12500	0.12500	0.0114
O	32*e*	.3m	0.23061	0.23061	0.23061	0.0156
[α = β = γ = 90º; *a* = *b* = *c* = 9.3176(7) Å and V = 808.931(1)Å³]; R_{wp} = 12.99%, R_p = 8.89%, R_{Bragg} = 4.76%, χ^2 = 1.42, *S* =1.18						
🡻Atoms	Wyckoff	Site	*x*	*y*	*z*	U [Å²]
Ag	16*d*	.-3m	0.50000	0.50000	0.50000	0.0211
Mo	8*a*	-43m	0.12500	0.12500	0.12500	0.0106
O	32*e*	.3m	0.23095	0.23095	0.23095	0.0188
[α = β = γ = 90º; *a* = *b* = *c* = 9.3183(9) Å and V = 809.11(1)Å³]; R_{wp} = 11.16%, R_p = 7.4%, R_{Bragg} = 4.55%, χ^2 = 1.25, *S* =1.12						
▼Atoms	Wyckoff	Site	*x*	*y*	*z*	U [Å²]
Ag	16*d*	.-3m	0.50000	0.50000	0.50000	0.0210
Mo	8*a*	-43m	0.12500	0.12500	0.12500	0.0114
O	32*e*	.3m	0.23064	0.23064	0.23064	0.0195
[α = β = γ = 90º; *a* = *b* = *c* = 9.3177(4) Å and V = 808.94(1)Å³]; R_{wp} = 12.73%, R_p = 8.54%, R_{Bragg} = 4.40%, χ^2 = 1.38, *S* =1.17						

β-Ag_2MoO_4 microcrystals synthesized with: ■ H_2O ($T_{boiling\ point}$ = 100°C); ● CH_4O ($T_{boiling\ point}$ = 65°C); ♦ C_2H_6O ($T_{boiling\ point}$ = 78.4°C); 🡻 C_3H_8O ($T_{boiling\ point}$ = 97.5°C) and ▼ $C_4H_{10}O$ ($T_{boiling\ point}$ = 118°C).