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Structural and Optical Characterization of Co_3O_4 Nanostructures Synthesized via Sol–Gel Method and Calcined at Different Temperatures

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INTRODUCTION & AIM

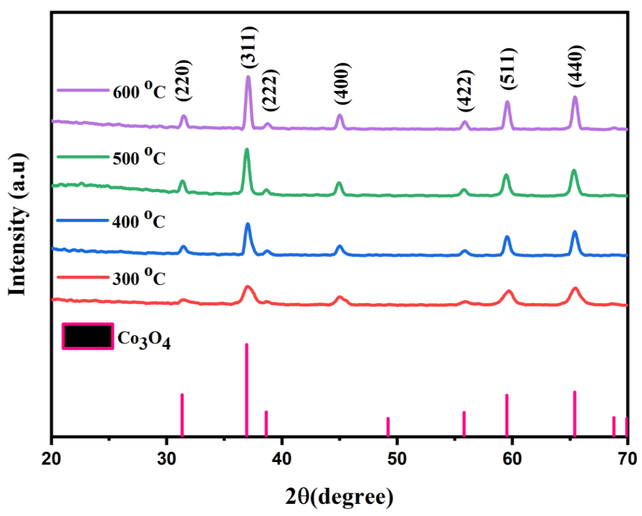
To synthesize cobalt oxide (Co_3O_4) nanoparticles using the sol–gel method and systematically investigate the effect of calcination temperature (300–600 °C) on their structural, thermal, and optical properties for potential applications in energy storage, photocatalysis, electrocatalysis, and environmental remediation. Cobalt oxide (Co_3O_4) is a mixed-valence transition metal oxide with a normal spinel structure, where Co^{2+} occupies tetrahedral sites and Co^{3+} resides in octahedral sites [1]. Among various synthesis routes, the sol–gel method provides advantages such as homogeneous precursor mixing, controlled particle growth, and improved phase purity. However, the influence of calcination temperature on the development of crystallinity, thermal stability, and optical characteristics of citric-acid-assisted sol–gel derived Co_3O_4 nanoparticles remains less explored [2,3]. This study addresses this gap by systematically examining Co_3O_4 nanostructures processed at different temperatures to understand their temperature-dependent structural evolution and functional behavior.

METHOD

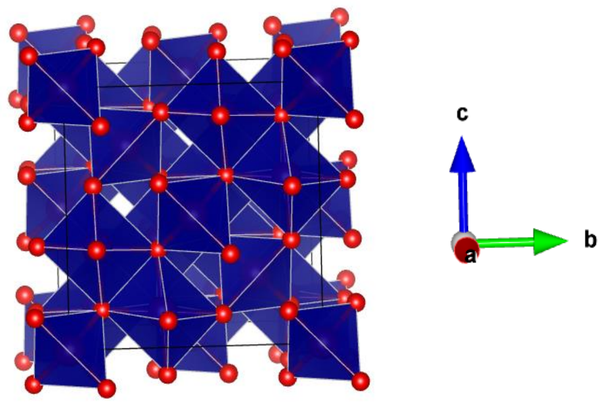
Cobalt oxide nanoparticles were synthesized using a citrate-assisted sol–gel method. Cobalt nitrate hexahydrate [$Co(NO_3)_2 \cdot 6H_2O$] and citric acid were dissolved separately in distilled water in a 1:1 molar ratio and mixed under continuous stirring. The resulting pink solution was heated at 100 °C for 2 hours to form a homogeneous sol, which gradually transformed into a gel during evaporation. The formed xerogel was dried at 130 °C for 2 hours to remove residual moisture and then calcined at 300, 400, 500, and 600 °C for 3 hours. After calcination, black Co_3O_4 nanoparticles were obtained, cooled to room temperature, ground using a mortar and pestle, and used for further characterization.

RESULTS & DISCUSSION

X-Ray diffraction:

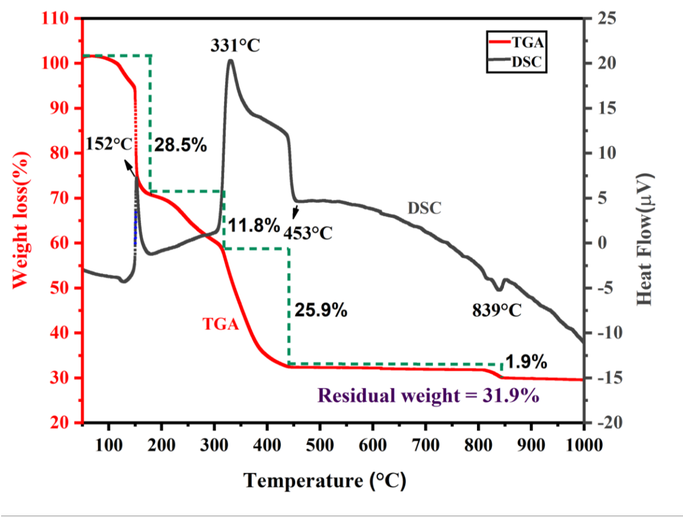


JCPDS Card No: 01 – 074 – 1656
Crystal Structure: Spinel Cubic phase
Space Group: Fd3m

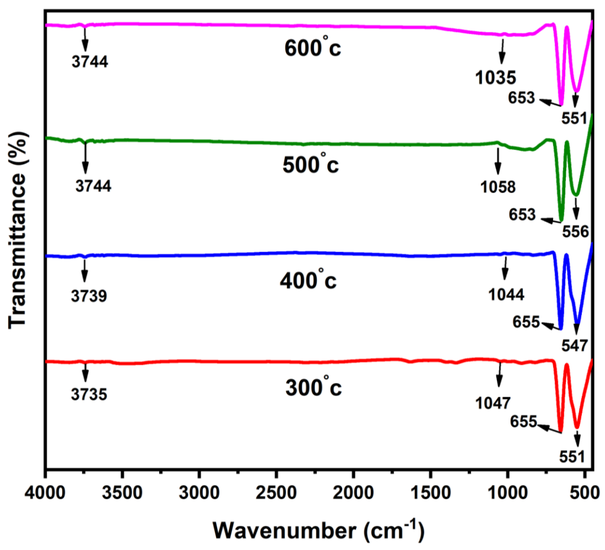


Temperature	Average crystallite size (nm)	Dislocation density δ (m^{-2})	Lattice parameter (Å)
300°C	15.373	0.0042	8.051
400°C	32.345	0.0009	8.057
500°C	33.948	0.0008	8.066
600°C	26.209	0.0014	8.062

TGA-DSC:

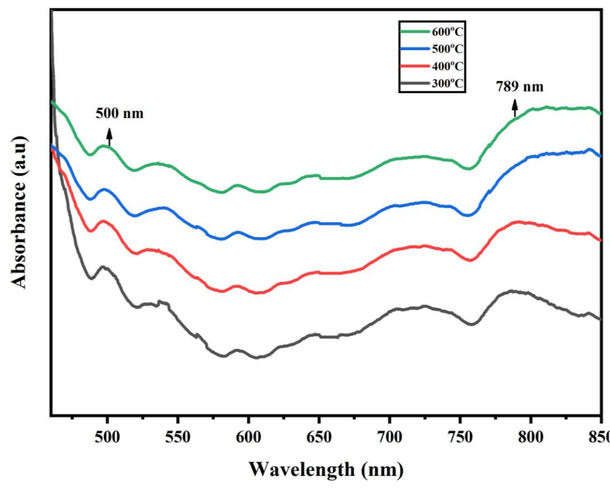


FT-IR Spectroscopy:



Bond assignment (functional group)	300 °C	400 °C	500 °C	600 °C
O-H vibrations	3735	3739	3744	3744
C-H vibrations	1047	1044	1058	1035
O-Co-O stretching vibrations	655	655	653	653
Co-O stretching vibrations	551	547	556	551

UV-Vis DRS:



Annealing Temperature	Band Gap Energy (eV)
300 °C	2.45
400 °C	2.44
500 °C	2.42
600 °C	2.43

CONCLUSION

Co_3O_4 nanoparticles were successfully synthesized and stabilized above 450 °C, forming a cubic spinel phase across 300–600 °C. Calcination temperature strongly influenced their microstructure and optical behavior, with crystallite size increasing up to 500 °C and slightly decreasing at 600 °C, while the band gap varied from 2.45 eV to 2.42 eV and rose marginally at higher temperature due to defect-related transitions. These results confirm that controlled calcination effectively tunes the structural and electronic properties of Co_3O_4, underscoring its strong potential for photocatalysis and energy-storage applications.

FUTURE WORK / REFERENCES

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2. Ceram. Int. 51 (2025) 11467–11479. https://doi.org/10.1016/j.ceramint.2025.01.001
3. Nanomaterials, 15, (2025) 932. https://doi.org/10.3390/nano15120932.