**ABSTRACT**

A higher capacity and better cyclability are apparent when [magnesium](https://www.sciencedirect.com/topics/chemistry/magnesium-atom) is introduced into the structure of LiCoO2 (y = 0.15). XRD analysis of LiMgyCo1−yO2 (y = 0, 0.1, 0.15), synthesized at 800 °C using a microwave assisted method, shows that the material is in the R-3m space group and to have a slightly expanded unit cell that increases with greater magnesium doping. Structural analysis by [X-ray absorption spectroscopy](https://www.sciencedirect.com/topics/materials-science/x-ray-absorption-spectroscopy) (XAS) at the Co K-edge, L-edge and O K-edge shows that the magnesium is located in the transition metal layer rather than in the lithium layer and the charge balance results from the formation of [oxygen vacancies](https://www.sciencedirect.com/topics/engineering/oxygen-vacancy) rather than Co4+, while cobalt remains in the 3+ oxidation state. Interestingly, oxygen is found to participate in the charge compensation. Both magnesium, in the transition metal layer, and the Co-defect structure are attributed to the contribution towards structural stabilization of LiCoO2, thereby resulting in its enhanced electrochemical performance.