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Positive electrode materials, LiMg*x*Ni0.5−*x*Co0.5O2 (*x* = 0 < *x* < 0.5), have been successfully synthesized by microwave-assisted solution technique. The precursor has been analyzed by TG/DTA and the powder was calcined at 850 °C. The XRD patterns reveal that the synthesized materials exhibit hexagonal layered structure corresponding to R3-m space group. Coin cells of 2016 type have been fabricated using the synthesized layered material as cathode active material and lithium foil used as counter and reference electrode. Test cells were operated in the potential limits between 2.7 and 4.3 V using 1 M LiPF6 in 1:1 EC/DEC as electrolyte. LiMg0.2Ni0.3Co0.5O2 material delivers an average discharge capacity of around 165 mA hg−1 at 0.1 C rate over the investigated 20 cycles.