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The development of sustainable, eco-friendly, cost-effective and renewable energy sources is required to address the increasing demand for energy. The rapid depletion of fossil fuels has also raised environmental concerns and has further pushed scientists to find an alternative source of energy. Hydrogen is one of the most appealing alternatives for future energy applications. At present, steam methane reforming method is used for large scale production of hydrogen which also consumes conventional fossil fuels and release greenhouse gases such as CO 2. The generation of hydrogen through water splitting has gained considerable researchers' attention due to zero carbon footprint as well as high gravimetric energy density of hydrogen. Hydrogen production from the electrolysis of water is one of the superior techniques owing to the production of high purity hydrogen at large scale without emission of greenhouse gases. The electrolysis of water is carried out in two half-reactions, which are hydrogen evolution reaction (HER) and oxygen evolution reaction (OER). The well-known equation for half-cell reaction of HER is $2H + (aq) + 2e - \rightarrow H2$ (g). Till date, Pt and Pd based materials has been regarded as the best electrocatalyst to carry out HER reaction as they require very low overpotential and gives very high current density. However, the main obstruction in their commercialization to produce large scale hydrogen is their high cost and scarcity. Therefore, developing cost-effective and highly efficient electrocatalysts based on non-noble metals for the hydrogen evolution reaction (HER) is critical to promote hydrogen generation at large scale to meet future energy demand. In the present thesis, we have developed a low-temperature process to synthesize ultrathin Co 2 P 4 O 12 and Ni 2 P 4 O 12 nanosheets. The Co 2 P 4 O 12 and Ni 2 P 4 O 12 nanosheets show an efficient catalytic activity towards HER and high stability due to presence of P 4 O 124- cyclic ring in acidic medium. Further, the catalytic activity of metal cyclotetraphosphate was enhanced by synergistic effect of bimetallic cyclotetraphosphates (CoNiP 4 O 12). To further enhance the activity of bimetalliccyclotetraphosphates, their composites with reduced graphene oxide (rGO) has also been explored to increase its surface area and electronic properties. We have also investigated transition metal phosphides for HER. In this, we have synthesized cobalt phosphide nanorods and iron phosphide nanoparticles. Co 2 P nanorods and FeP nanoparticles shows an excellent activity towards HER in the acidic media. In summary, this thesis focuses on stabilization of various phosphorous based materials and demonstrates the potential of their electrocatalytic properties. The synthesized materials showed promising catalytic activity towards hydrogen generation.

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