Development of suitable technologies for the production of alternate source of energy has become a daunting task in the recent decades. The depletion of fossil fuels and increasing population further increased these complexities. Biodiesel, a suitable alternative to conventional energy system has attracted the attention of the scientific community. Though there are many ways for biodiesel production, transesterification has gained a lot of importance due to renewability, higher cetane number and combustion efficiency. Transesterification is often performed in presence of catalyst (homogeneous, heterogeneous and enzymatic) and development of these catalysts (heterogeneous) from industrial wastes with high basic properties would help in subsiding pollution. Though this flyash is being utilized in brick manufacture, agricultural fertilizer and substituted upto 66% of cement in construction of dams, an effective way of utilizing this waste so that it can be converted into a value added product is one of the daunting challenges faced by the scientific community. Utilization of flyash for synthesis of different catalysts in biodiesel production might have scope for further research. This work presents the use of flyash as starting material for the synthesis of heterogeneous catalyst for transesterification of mustard oil. The entire work carried out in this research is divided into five major parts: (a) Physico-chemical properties and thermal degradation studies of commercial oils in nitrogen atmosphere. (b) Utilization of flyash as heterogeneous catalyst for transesterification. (c) Selective preparation of zeolite X and A from flyash and its use as catalyst for biodiesel production. (d) Preparation and characterization of hydrotalcite like materials from flyash for transesterification. (e) Thermal stability, physical properties, chemical composition and degradation kinetics of biodiesel: A Comparative study.