The infrastructural development is one of the essential aspect for the betterment of social well-being of human race. Construction and building materials plays a major role in the development of infrastructure. Concrete, the most widely accepted construction and building material faces several technological, economical and environmental problems because of the use of ordinary Portland cement (a non-sustainable material) as the binding agent. Partial or complete replacement of OPC with supplementary cementitious materials in concrete constructions can provide major reductions to these problems. The deterioration of concrete structures in the marine environment is a growing problem in Gulf countries. The incorporation of volcanic ash as a supplementary cementitious material in blended cement based concrete has attracted much attention because of its better performance in terms of strength and durability due to pozzolanic action of volcanic ash in marine environment.

Quantitative and qualitative analysis of mineralogical phases formed during cement hydration process using XRD/Rietveld analysis is emerging as a widely accepted tool in cement and concrete research. Evolution of different cement hydration products are monitored by X-ray powder diffraction measurements as a function of time. In this paper, we present the early age hydration behavior of cement paste samples prepared by partially replacing OPC with regionally available volcanic ash, monitored both qualitatively and quantitatively by using X-Ray Fluorescence Spectroscopy (XRF) and X-Ray Diffraction (XRD) spectroscopy. The internal standard method is adopted for the absolute phase quantification and estimation of the amorphous or non-identified phase contents by XRD analysis. Topas Academic V4.0 software is used for Rietveld refinement. Qualitative X-ray diffraction analysis showed the presence of portlandite (CH), a broad hump of C-S-H between 28 and 32.2 two theta unit along with traces of alite (C3S) and belite (C2S) phases. Ettringite (Ca6Al2(SO4)3(OH)12·26H2O), brownmillerite (Ca2(Al,Fe)2O5), and anorthite (CaAl2Si2O8) were also identified in minor amounts after 28 days of curing.

**Key Words:** Supplementary cementitious material, Cement Hydration, Volcanic Ash, Rietveld Analysis.